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INFAC

Management and Operation Of the DoD Instrumented Factory for Gears

Quench Press Hardening with Low Copper Plating

March 28, 2001

U. S. Army Aviation and Missile Command
Redstone Arsenal
AMSAM-RD-SE-MT
Huntsville, Alabama 35898-5270

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13. ABSTRACT (Maximum 200 words) Copper plating is used as a carbon stop-off during the carburization and austenitization operations associated with the manufacture of precision gears. The “Low Copper” project's goals were to optimize the thickness of copper needed during carburization, and to eliminate the need to re-plate copper prior to austenitizing, via atmosphere control in a rotary furnace. Traditionally, plating that is 0.001 to 0.003 inch thick is used as a carbon stop-off. Experimentation was aimed at reducing this thickness to between 0.0001 to 0.0005 inch, roughly an order of magnitude thinner. Three designed experiments were performed by IIT Research Institute. Honeywell Engines & Systems of Phoenix, AZ followed the experimentation at IIT Research Institute by performing preproduction tests and additional experiments. Honeywell also prepared a cost benefit analysis to detail and document the projected process savings that could be gained by implementation of the technique. An industry-wide cost-benefit analysis and implementation plan is included. Annual savings realized by pervasive implementation of the technique were estimated at over $1,000,000 per year.

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Contents

Abstract

Copper plating is used as a carbon stop-off during the carburization and austenitization operations associated with the manufacture of precision gears. The “Low Copper” project's goals were to optimize the thickness of copper needed during carburization, and to eliminate the need to re-plate copper prior to austenitizing, via atmosphere control in a rotary furnace. Traditionally, plating that is 0.001 to 0.003 inch thick is used as a carbon stop-off. Experimentation was aimed at reducing this thickness to between 0.0001 to 0.0005 inch, roughly an order of magnitude thinner. Three designed experiments were performed by IIT Research Institute. Honeywell Engines & Systems of Phoenix, AZ followed the experimentation at IIT Research Institute by performing preproduction tests and additional experiments. Honeywell also prepared a cost benefit analysis to detail and document the projected process savings that could be gained by implementation of the technique. An industry-wide cost-benefit analysis and implementation plan is included. Annual savings realized by pervasive implementation of the technique were estimated at over $1,000,000 per year.

Executive Summary

Copper plating is used as a carbon stop-off during the carburization operation associated with the manufacture of precision gears. It is also used as a diffusion barrier during the austenitization operation associated with the press quenching of precision gears. This project's goals were to optimize the thickness of copper needed during carburization, and to eliminate the need to re-plate copper prior to austenitizing, via atmosphere control in a rotary furnace. Traditionally, plating that is 0.001 inch to 0.003 inch thick is used as a carbon stop-off. Experimentation was aimed at reducing this thickness to between 0.0001 inch and 0.0005 inch, roughly an order of magnitude thinner.

To accomplish this goal, three experiments were performed. The first experiment investigated the ability of thin copper plating to successfully stop-off carbon during a typical carburization cycle. The second experiment explored further and attempted to use thin copper during the entire heat treating cycle. The third experiment was used to verify the results of the first two and to explore new variables of interest brought out by the initial experimentation.

Honeywell Engines & Systems of Phoenix, AZ followed the experimentation at IIT Research Institute by performing preproduction tests and additional experiments. Honeywell also prepared a cost benefit analysis to detail and document the projected process savings that could be gained by implementation of the technique.

The problem was modeled using Fick's diffusion laws. Diffusion of carbon through the copper matrix was identified as an unlikely failure point for the stop-off. However, porosity in the plating was identified as the most likely route for carbon diffusion into the steel substrate.

A cost-benefit analysis was performed to gauge industrial savings through implementation of the technique. Manufacturers that use selective copper plating to produce components were contacted by telephone and interviewed. A savings matrix was produced from these interviews. Total savings were estimated at over $1,000,000 per year.

An implementation plan, detailing steps that a manufacturer would have to perform to implement the technique, was developed. This technology has the benefit of reducing the amount of copper anode used, reducing time spent in copper plating baths, and eliminating mid-process cleaning, stripping and re-plating operations and their associated cleaning operations.

This processing technique has application to the manufacture of any selectively carburized component composed of SAE/AISI 9310H steel. U.S. Army weapon systems using components of these types include the Black Hawk and Apache Longbow rotorcraft, and the F-117A, F-22, B-1B, and F-15 fixed-wing aircraft, among many others.

The analyses performed indicated that copper plating that is 0.0005 inch thick can successfully be used to stop-off carbon in a process cycle comprised of a 1700°F carburization operation and a 1525°F austenitization operation under an protective atmosphere.

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Technical/Manufacturing Methods Report

Introduction

Precision gears used in military helicopter powertrains are composed of steel that must be heat treated to impart necessary material properties for peak performance. Currently, the most widely used steel in these powertrains is SAE/AISI 9310H. The chemical composition of SAE/AISI 9310H is listed in Table 1.

Properties that these gears must possess include a hard wear surface, high fracture toughness in both the surface and the core, and high strength. A hard surface is needed to prevent wear of mating surfaces in a gear train. High surface fracture toughness is needed to prevent cracking of the surface and the associated lowered performance, accelerated degradation of the part and mating parts, and promotion of through-cracking of the part. High core fracture toughness is necessary to prevent through-cracking of the part. High strength is needed to carry the tremendous loads demanded by the military application.

The aforementioned properties are usually imparted into a steel surface and core in the presence of a controlled-composition gas or atmosphere via deliberate heating and cooling cycles. The generalized heat treatment cycle for SAE/AISI 9310H used in precision gears can be found in Figure 1.

Each step in the process illustrated in Figure 1 can be broken down further. Additionally, there are intermediate steps that are not mentioned. The process described is a general overview of what needs to be done to a gear to impart beneficial properties into it. It does not describe the specific process for any particular component. To fill this gap and to narrow the focus of this paper, a process is now described that applies to a specific class of components: selectively carburized, press-quenched precision gears.

Table 1 - Chemical composition of SAE/AISI 9310H steel [Tim96].

Element	Weight % (low/high)
C	0.07/0.13
Mn	0.40/0.70
Cr	1.00/1.45
Ni	2.95/3.55
Mo	0.08/0.15
Si	0.15/0.35
P	0.035 max
S	0.040 max
Cu	0.350 max
Fe	Balance
Other	…

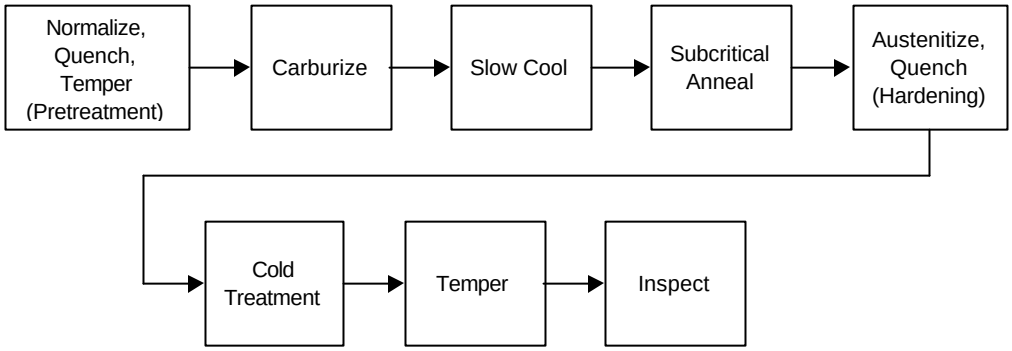


Figure 1 – The general cycle for the Heat Treatment of SAE/AISI 9310H.

Selective carburization is the process by which specific areas of the surface of a component are carburized, while others are not. The carburized surface is conventionally referred to as the “case”. Carbon that would normally be diffusing into the substrate during carburization is “stopped-off”, or prevented from diffusing into chosen areas. There are two major reasons for performing selective carburization:

1) Minimization of distortion during the manufacturing process.
2) The alteration and control of surface toughness of selected areas of the gear for the benefit of in-service properties.

Both reasons fall into the subjects of manufacturing and design-for-service.

Distortion during heat treatment can be defined as any change in geometry of the incoming component. Distortion can occur during manufacturing due to the following fundamental causes [TH97]:

1) Relief of residual stresses
2) Thermal expansion/contraction
3) Phase transformations

Distortion due to phase transformations is what the process of selective carburizing tries to prevent. By driving carbon into a surface, the surface will expand due simply to the increased carbon content. It will also expand if the surface is allowed to cool quickly, as in quenching.

A volumetric increase is associated with the transformation from austenite, which is stable at and above about 1475°F (for an SAE/AISI 9310H surface with approximately 0.85% carbon), into martensite. Because this surface has boundary conditions that prevent it from expanding only outwards into free space, residual stresses (usually compressive in the vicinity of the surface) form in the case as well as any surrounding volume of non-carburized surface and core. These stresses, depending on the design geometry of the part, can cause a change in the final geometry. One can control the extent of the distortion by indirectly controlling the amount of expansion. This is accomplished by carefully selecting the surface area that it is necessary to carburize.

This process of carburizing only selected areas of the surface has the added benefit of retaining the fracture toughness of the non-carburized surfaces. The retention of fracture toughness is due the formation of low-carbon martensite rather than high-carbon martensite in these areas during subsequent austenitizing and quenching.

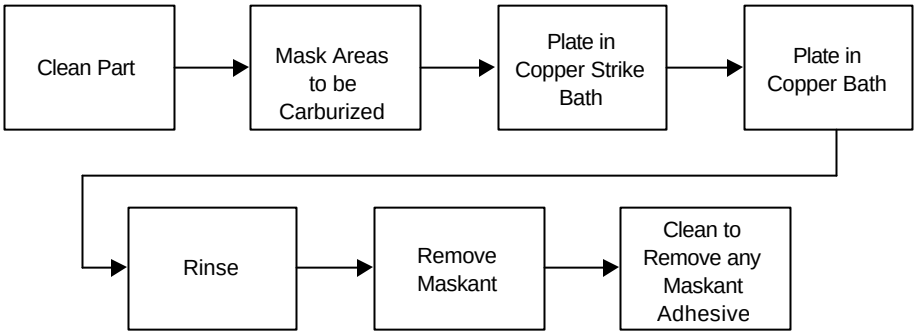
Press quenching is a process in which an austenitized part is placed in a quenching apparatus that constrains the part between 2 pulsating dies while at the same time performing an oil quench via specially designed channels within the die. The purpose of the die is to fix the position of the part when phase transformations occur so that distortion is minimized.

Together, the processes of selective carburizing and press quenching provide for a method to minimize distortion and create both hard surfaces and surfaces with high fracture toughness. The two processes can obviously also be used separately. For example, a selectively carburized part can be carburized, direct quenched (rather than slow cooled, austenitized and quenched) and tempered. Alternatively, a part can have all of its surface area carburized and be press quenched to minimize distortion.

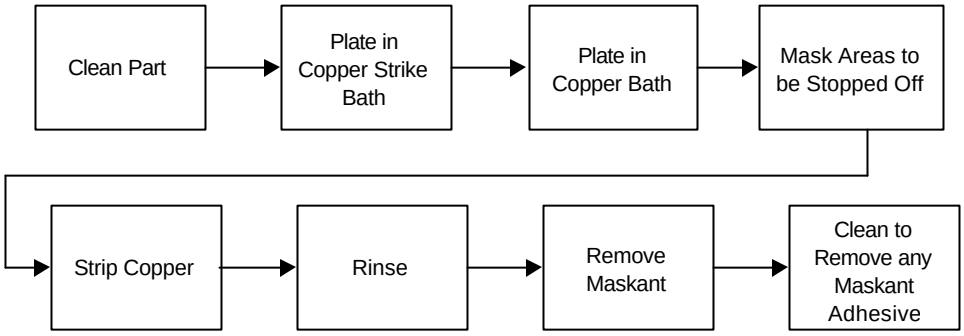
There are two methods by which steel surfaces may be selectively carburized [INFAC95]. One uses stop-off paint [Pre98], and the other uses copper plating. Stop-off paint consists of refractory substances and a binder of glass that can be brushed onto the surface of a part where carbon is to be stopped-off. Cyanide-based copper plating is used as a stop-off material because of the low solubility and diffusivity of carbon in copper. Unfortunately, precise quantification of the diffusivity of carbon in copper is not readily available in published literature, which makes modeling and prediction of the system difficult [HKK88]. Diffusion of carbon through copper is known however to be small for typical carburization cycles. This modeling problem is further discussed in a subsequent section of this paper.

Copper plating via cyanide-based baths is generally a twofold process. First a copper strike is applied. The copper strike applies quickly and adheres easily to the surface, but cannot apply thicknesses greater than about 0.0001 inch because layer growth drops as the thickness increases. Secondly, the part is placed in a copper bath that has the capability of applying thicker coats, usually up to a few thousandths of an inch. Pictures of gears plated to typical copper plating thickness are shown in Figure 2. A more detailed version of the process is broken down in Figure 3.

Figure 2 – Gears that have been copper plated to (a) 0.001-0.003 inch selectively and (b) 0.0003-0.0005 inch non-selectively.



(a)



(b)

Figure 3 - Two methods of applying a cyanide-based selective copper plate to a steel substrate. (a) Mask and plate. (b) Plate, mask and strip.

Problem Statement

To reduce the cost of the manufacture of precision gears it would be beneficial to reduce or eliminate intermediate process steps. The carburization process for SAE/AISI 9310H steel was summarized in Figure 1. By choosing to manufacture a gear using selective carburization, additional process steps must be added to the process. Figure 4 is an outline of the selective carburization process for SAE/AISI 9310H.

The intermediate processing steps of washing, grit blasting, stripping and re-plating add considerable cost to the process. It is desirable to find a way to either eliminate or minimize the cost associated with the additional processing steps. To examine the problem more closely, one must understand the reasons associated with the steps in the current process. Table 2 provides a concise breakdown of the selective carburization process and reasons for each step.

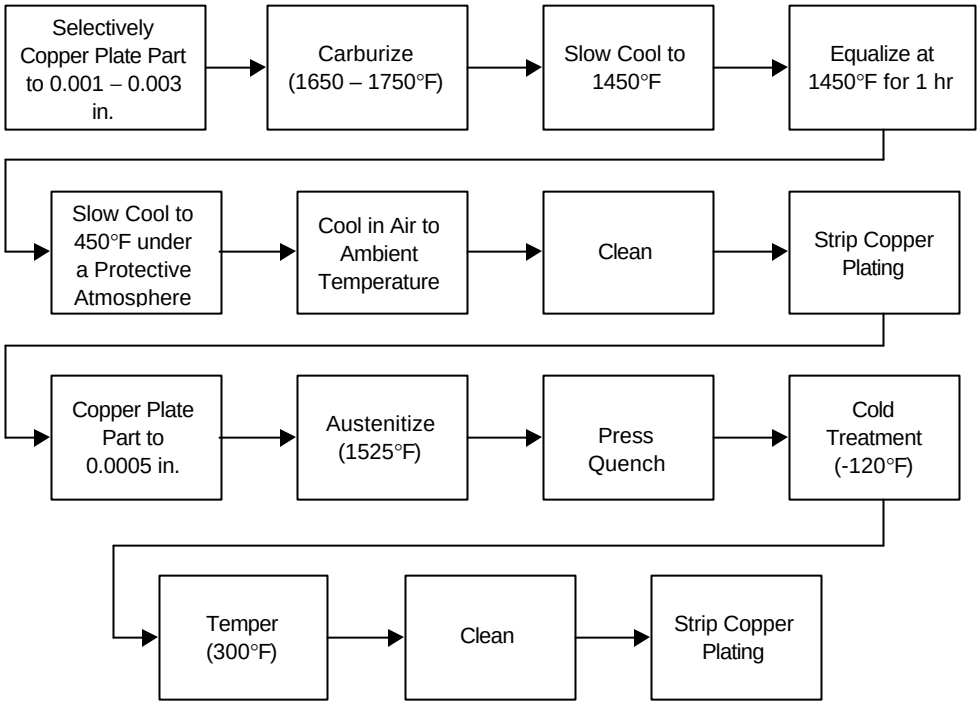


Figure 4 - A generalized selective carburization process for SAE/AISI 9310H.

Table 2 - Breakdown of the conventional selective carburization process, with emphasis on the steps in the process which define it as “selective”.

Step	Reason
Selective Copper Plate	Apply carbon stop-off (0.001 to 0.003 in thick) to not-to-carburize surface areas
Carburize	Diffuse carbon into selected surfaces
Slow Cool to 1450°F	For machinability of any proud surfaces
Equalize	To minimize distortion and residual stress development during cooling
Slow Cool to 450°F	Slow cool under endothermic atmosphere for surface protection
Cool to Room Temp	Cool for subsequent operations
Clean	Remove surface contaminants
Strip Copper Plating	Remove stop-off
Copper Plate	Re-plate all-over prior to austenitizing for carb/decarb/oxidation protection and to hold necessary tolerance during press quenching (0.0005 inch thick)
Austenitize	Austenitize for quenching (Usually in a rotary furnace for press-quenched parts
(Press) Quench	Quench to harden the material
Cold Treat at -120°F	Cold treat to maximize austenite-to-martensite transformation
Temper at 300°F	Temper for toughness
Clean	Preparation for copper stripping
Strip copper	Remove surface protection

So, copper plating is reapplied after carburizing for three major reasons:

1) To prevent carburization/decarburization during austenitizing and subsequent operations.
2) To prevent intergranular oxidation (IGO) of surfaces during austenitizing and subsequent operations.
3) To hold tolerance during press quenching as tight as possible. The copper plating can be viewed to add 0.0005 inch on either side of a parallel-axis gear. Contrast this with the 0.001 to 0.003 inch that would be added to the tolerance if the initial selective plating would still have been in use on the surface of the part.

The third point highlights the need to re-plate the copper at a thinner thickness prior to hardening. It is done to allow the manufacturer to produce a part with greater tolerance

control; The copper plating, especially at thicknesses on the order of a few thousandths on an inch, is not necessarily of uniform thickness. These cleaning, stripping and re-plating operations add additional cost to the product. It would be desirable if these steps did not have to be performed at all. To accomplish this one must show that:

1) Thin copper plating (0.0005 inch) is capable of stopping-off carbon in the carburizing process. (Reason 3)
2) The carbon level of exposed surfaces and copper plated surfaces are not affected in the austenitizing and subsequent operations. The atmosphere in the austenitizing furnace needs to be protective in order to accomplish this. (Reason 1)
3) Oxidation of the exposed surfaces and copper plated surfaces can be adequately controlled. The atmosphere in the austenitizing furnace needs to be protective in order to accomplish this. (Reason 2)

Purpose and Goal

The purpose of this project was to examine the effectiveness of cyanide-based thin copper plating as a carbon stop-off during the selective carburizing process of AISI/SAE 9310H steel. Further, the purpose was to examine the factors affecting the level of surface carbon in the austenitizing furnace, specifically, the effect of the carbon potential of the atmosphere in the austenitizing furnace.

The goal of the project was to show that thin copper plating (between 0.0003 and 0.0005 inch thick) could be used to selectively carburize a part, and that no further plating operations would be needed to successfully complete the heat treatment of the part. [HT92] has stated that 0.0005 in plating is adequate to stop-off carbon as long as the copper is of a fine grained form, demonstrating that a thin (or “low”) copper technique is feasible.

Project Methodology

There were six major steps in the technology development:

1) State of the Art Review (SOAR)
2) Design of Experiment 1 (DOE 1)
3) Design of Experiment 2 (DOE 2)
4) Design of Experiment 3 (DOE 3)

5) Preproduction tests at Honeywell Engines & Systems[1]
6) Construction of a Diffusion Model

The purpose of the SOAR was to examine current industry practice in the field of selective carburizing. It was composed of three elements: a literature search, an industry benchmark and a report.

DOEs 1, 2 and 3 each examined variables thought to affect the properties of the tested parts. DOE 1 was used as a screening experiment for an isolated carburization heat treatment. The purpose of a screening experiment is to "screen" out what the major factors are in a process. DOE 2 was also used as a screening experiment, in this case, as a screen for the whole heat treatment process. DOE 3 then was used to refine the factors and levels in DOE 2 and to verify the findings of DOE 2.

The pre-production tests at Honeywell served the purpose of moving from a laboratory-scale experiment to a production-scale experiment. Honeywell provided a test bed for the thin copper technique. Honeywell also provided the copper plating used in IITRI's experimentation.

The diffusion system was modeled as part of the project to gain a better understanding of the mechanisms that may be playing a role in the copper plating's failure.

State of the Art Review

The SOAR report is included herein as Appendix A. A summary of the research leading to the report follows.

The SOAR, comprised a literature search, an industry benchmark and a report. It was conducted at the very beginning of the project before any experimentation began. The report was to serve as a benchmark for further technological development.

A literature search was conducted to see if there was any published literature on the following topics:

1) Selective Carburizing
2) Heat Treatment Processes of SAE/AISI 9310H
3) Press Quenching
4) Copper Plating Processes

[1] Honeywell Engines & Systems will simply be referred-to as "Honeywell" for the remainder of this report for brevity.

The industry benchmark involved visits to selected manufacturers and tours of their plating and heat treating operations. Visits were made to Bell Helicopter Textron in Fort Worth, Texas, Rolls-Royce Allison Engines in Indianapolis, Indiana and Honeywell Engines & Systems in Phoenix, Arizona

A report was then generated based on the results of the literature search and industry benchmark.

Experimentation

DOE 1

Overview

The purpose of DOE 1 was to test whether thin copper plating could be used to effectively stop-off carbon during a typical SAE/AISI 9310H steel carburization cycle. The goal was to show that thin copper plating in the thickness range of 0.0001 to 0.0005 inch could be used for this purpose.

Discussion between IITRI and Honeywell personnel led to the following variables being examined in the experiment:

1) Copper Plating Thickness
2) Carburizing Temperature
3) Case Depth / Time in the Furnace
4) Surface Finish
5) Handling

These factors were deemed to have the most influential affects on the final output of the selective carburizing process. Copper plating thickness was an obvious choice as a factor to examine. The choices of the other factors revolved around what IITRI and Honeywell personnel thought may influence failure of the plating. Failure, in this sense, is the penetration of carbon through the copper stop-off and into the steel substrate. Carburizing temperature was chosen as a blocking factor. Blocking allows one to examine a range of process levels that the proposed process will be exposed to in a real production situation, but are not believed to have different influences at different levels.

The factors “Case Depth” and “Time in the Furnace” were considered a single variable in the experiment. The reason for this is because, in general, parts requiring shallower case depths are processed at lower temperatures for greater control of the case depth. Processing them at the lower temperature requires carburizing them for a longer period of time due to the lower mobility of carbon at lower temperatures.

Surface finish and handling are more properly referred to as noise variables because they are not easily or cost-effectively controlled during the manufacturing process.

Once the factors were chosen, levels were set for each of them:

1) Copper Plating Thickness
    a) 0.0001 in

b) 0.0003 in
c) 0.0005 in

2) Carburizing Temperature
   a) 1650°F
   b) 1750°F
3) Case Depth / Time in the Furnace
   a) 1650F
      i) 0.010 in Case Depth
      ii) 0.030 in Case Depth
   b) 1750F
      i) 0.030 in Case Depth
      ii) 0.050 in Case Depth
4) Surface Finish
   a) Smooth (Incoming material surface)
   b) Rough (Machined surface)
5) Handling
   a) Careful
   b) Rough

The resultant test matrix can be found in Table 3.

Again, examination of the factors/levels reveals that shallower-cased parts were processed at the lower carburizing temperature.

Table 3 - DOE 1 experimental matrix.

Sample Number	Plating Thickness [in]	Case Depth [in] / Temperature [°F]	Careful / Rough Handling
01	0.0001	0.030/1650	R
02	0.0003	0.010/1650	C
03	0.0001	0.050/1750	R
04	0.0005	0.010/1650	R
05	0.0003	0.030/1650	R
06	0.0005	0.030/1650	R
07	0.0001	0.010/1650	C
08	0.0005	0.030/1750	C
09	0.0005	0.030/1750	R
10	0.0005	0.050/1750	C
11	0.0001	0.010/1650	R
12	0.0003	0.050/1750	R
13	0.0001	0.030/1750	R
14	0.0005	0.030/1650	C
15	0.0005	0.010/1650	C
16	0.0003	0.030/1650	C
17	0.0001	0.050/1750	C
18	0.0001	0.030/1650	C
19	0.0003	0.010/1650	R
20	0.0005	0.050/1750	R
21	0.0003	0.050/1750	C
22	0.0001	0.030/1750	C
23	0.0003	0.030/1750	C
24	0.0003	0.030/1750	R

Experimental Procedure

Twenty-four samples composed of SAE/AISI 9310H were acquired from Honeywell. The geometry is illustrated in Figure 5. Each was assigned a sample number and had the number stamped on one end. Rough surfaces were then created on the samples via a CNC lathe. A burr created by this machining operation was left on the sample because it's removal could have caused damage to either or both of the surfaces of interest. The surface roughness of the samples was measured at three stages in the experiment:

1) After creating rough surfaces, but prior to plating.
2) After plating.
3) After heat treatment.

Surface roughness was measured on both the as-received end and the turned end. Three observations were made at each stage, on each sample. Mass of the samples was measured at the same process points as surface roughness. Mass was examined to gain a better understanding of how much copper plating was being deposited on the surfaces of the samples. One measurement was made on each sample.

These samples (and the subsequent sample sets in DOEs 2 and 3) were processed using equipment at the Department of Defense (DoD) Instrumented Factory for Gears (INFAC), operated by IIT Research Institute (IITRI). Parts were carburized in a modified Lindberg gas-fired vertical radiant tube furnace.

There were four furnace loads in this experiment:

1) 1650F, 0.010 in Case Depth, 0.85% Carbon Potential
2) 1650F, 0.030 in Case Depth, 0.85% Carbon Potential
3) 1750F, 0.030 in Case Depth, 0.85% Carbon Potential
4) 1750F, 0.050 in Case Depth, 0.85% Carbon Potential

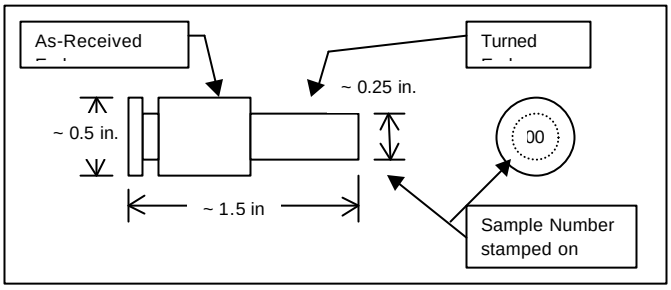


Figure 5 - Geometry of DOE 1 samples. In DOE 1, each of the samples was copper plated according to the experimental matrix in Table 3. Each sample was copper plated over 100% of their surface area in this experiment according to the test matrix in Table 3.

The parts were heat treated as follows:

1) The parts were racked according to a randomized design; the parts were hung using copper wire
2) The furnace was brought to temperature
3) The carbon potential was set and allowed to stabilize
4) The parts were introduced into the furnace heat zone
5) The parts were carburized for a prescribed amount of time
6) The parts were brought into the furnace vestibule and allowed to cool to approximately 500°F
7) The parts were brought out of the vestibule and allowed to finish cooling

Surface Roughness Analysis

Rough surfaces were generated using a CNC lathe. The surface roughness of the samples, on both the as-received and turned ends, was measured using a TSK profilometer. Three readings were taken on each surface along the axial direction. The statistic of interest was R_a. The profilometer was previously calibrated by factory technicians. Calibration was checked via a calibrated testing block. The raw data can be found in Table B1 (Appendix B). Statistics from these data are displayed in Table 4.

Data was also gathered on each of the samples after they had been copper plated. The raw data for these measurements can be found in Table B2 (Appendix B). Statistics from these data are displayed in Table 5.

A slight increase in surface roughness was observed after copper plating.

Mass Analysis

The mass of each of the samples was measured at the three process points mentioned before. The raw data obtained can be found in Table C1 (Appendix C). The mass gain after plating, i.e. the increase in sample mass due to copper plating, is displayed in Table 6. The mass gain after carburizing was analyzed and found to be below the measurement capability of the system. The data obtained, however, did seem to indicate a positive relationship with increasing time in the furnace.

Macroetch Analysis

After heat treatment, surface roughness analysis and mass analysis, a macroetch was performed to check for carbon on the surface of the parts. The procedure used can be found in Table 7. No evidence was found for carbon penetration. Some spots that were found on the surface were determined to be due to contact with the rack during carburizing. The samples were originally hung, but the copper wire holding them did creep during the cycle.

Additionally, a “skirt” of discoloration surrounded the burr on a number of the samples. This was believed to have been caused by trapped etchant that was not washed away during the final rinse of the samples. The “skirt” itself developed after the samples sat for a few minutes on the examination table. Based on the visual inspection, there was no substantial evidence that carbon leaked on any of the specimens.

Microstructural Analysis

A microstructural examination was conducted to cross-check the results of the macroetch inspection. The analyzed samples were chosen based on their performance in the macroetch; and the poorest performing samples were chosen for analysis. The specific samples chosen were 1(TE), 3(AE), 4(TE), 8(TE), 9(AE), 10(AE), 12(AE), 12(TE), 13(AE), 16(AE), 18(TE), and 24 (TE).[2] The samples were each polished to a 3 μm finish and etched with 2% nital for approximately 3 to 5 sec. A total of 12 samples were examined, making this a 25% inspection. Recall that these samples were carburized at either 1650 or 1750 °F and furnace cooled.

Based on the photomicrographs taken, no evidence of carbon penetration was found on any of the specimens. This confirms the conclusion of the macroetch performed earlier. Representative micrographs can be found in Figure 6.

DOE 1 Conclusions

The main conclusion drawn from the analyses performed on the DOE 1 specimens was that carbon did not penetrate the copper layers of any of the plating thicknesses for the levels of the factors chosen.

That said, DOE 2 (the second screening experiment) took the experimentation a step further and examined the performance of thin copper throughout an entire heat treatment cycle.

Table 4 - Statistics from the DOE 1 surface roughness analysis (prior to copper plating). Reported values are R_a and are in μinch.

	As-received End	Machined End
Min	23.16	93.69
Max	64.34	124.09
Average	45.67	103.27

[2] TE refers to “turned end”, AE refers to “as-received end”.

Standard Deviation	8.58	9.65

Table 5 - Statistics from the DOE 1 surface roughness analysis (after copper plating). Reported values are R_a and are in µinch.

	As-received End	Machined End
Min	26.02	93.91
Max	62.64	132.08
Average	47.34	106.78
Standard Deviation	7.70	11.06

Table 6 - Mass gain of each of the DOE 1 samples after plating.

Plating Thickness (in)	Average Mass Gain (g)
0.0001	0.03
0.0003	0.15
0.0005	0.17

Table 7 - Macroetch procedure to check for carbon leakage.

Step	Action
1	Wash part thoroughly using soap and water
2	Rinse with water
3	Rinse with ethanol
4	Dry
5	Etch in 5% Nital for 30 seconds
6	Rinse in water
7	Etch in 13% HCl (ethanol base) for 30 seconds
8	Rinse thoroughly in water
9	Rinse with ethanol
10	Dry

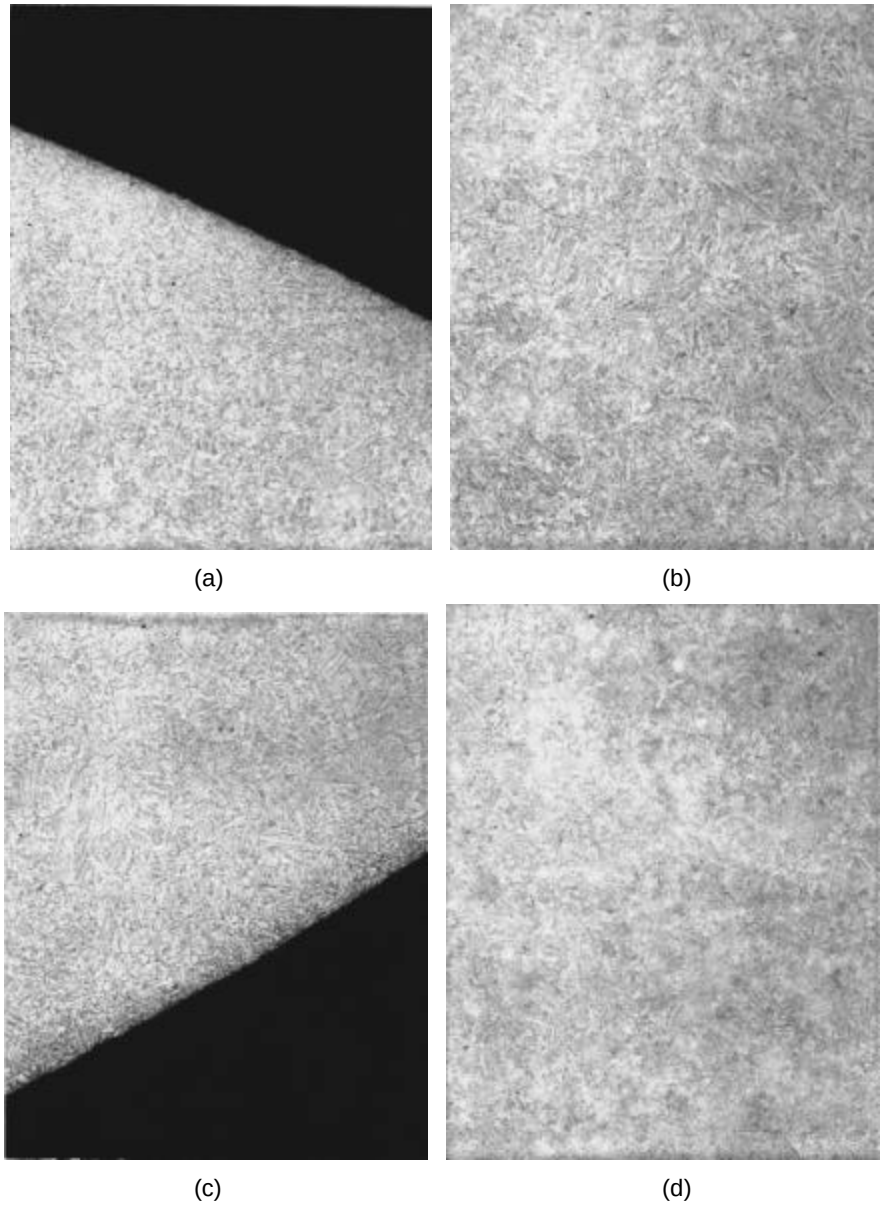


Figure 6 - Representative micrographs from the DOE 1 microstructural analysis. (a) Surface microstructure of sample number 4(TE). (b) Core microstructure of sample number 9(AE). (c) Surface microstructure of sample number 12(TE). (d) Core microstructure of sample number 24(TE). All micrographs were taken at 200x magnification.

DOE 2

Overview

The purpose of the second experiment was to examine the performance of thin copper that had gone through an entire simulated heat treatment cycle.

The goal was to show that thin copper plating is capable of stopping off carbon during an entire heat treatment cycle. As before, discussion between IITRI and Honeywell personnel led to the choices of factors and levels examined in the experiment. The choices were:

1) Case Depth
   a) 0.030 in, 1650°F, 0.85% Carbon Potential
   b) 0.050 in, 1750°F, 0.85% Carbon Potential
2) Copper Plating Thickness
   a) 0.0001 in
   b) 0.0005 in
3) Rotary Furnace Carbon Potential (Austenitizing Operation)
   a) 0.75%
   b) 0.95%
4) Austenitizing Time (Time in the Rotary Furnace)
   a) 30 min
   b) 90 min
5) Quench Transfer Time
   a) 10 sec
   b) 30 sec
   c) 50 sec

The last factor was considered a noise factor[3]. The corresponding experimental matrix can be found in Table 8.

The sample geometry was similar to that of DOE 1 except that new surfaces were not machined onto the samples. DOE 2 sample geometry can be found in Figure 7.

[3] A noise factor is a factor that is either too difficult or too costly to control during normal production, but may contribute to variation in a process.

Table 8 - DOE 2 experimental matrix. (continued on next page)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Rotary Furnace Carbon Potential (%)	Austenitizing Time (min)	Quench Transfer Time (sec)
1	0.0001	0.030/1650	0.75	90	30
2	0.0001	0.030/1650	0.95	30	10
3	0.0001	0.030/1650	0.75	90	50
4	0.0001	0.050/1750	0.75	30	50
5	0.0001	0.050/1750	0.95	90	10
6	0.0001	0.050/1750	0.95	30	10
7	0.0001	0.030/1650	0.75	30	30
8	0.0001	0.030/1650	0.95	90	50
9	0.0001	0.030/1650	0.75	30	10
10	0.0001	0.050/1750	0.95	30	30
11	0.0001	0.030/1650	0.95	30	30
12	0.0001	0.030/1650	0.95	90	10
13	0.0001	0.050/1750	0.95	30	50
14	0.0001	0.030/1650	0.75	30	50
15	0.0001	0.050/1750	0.75	30	30
16	0.0001	0.050/1750	0.95	90	30
17	0.0001	0.050/1750	0.75	90	10
18	0.0001	0.030/1650	0.75	90	10
19	0.0001	0.050/1750	0.75	90	50
20	0.0001	0.050/1750	0.75	90	30
21	0.0001	0.030/1650	0.95	30	50
22	0.0001	0.030/1650	0.95	90	30
23	0.0001	0.050/1750	0.95	90	50
24	0.0001	0.050/1750	0.75	30	10

Table 8 - DOE 2 experimental matrix. (concluded)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Rotary Furnace Carbon Potential (%)	Austenitizing Time (min)	Quench Transfer Time (sec)
25	0.0005	0.050/1750	0.95	30	30
26	0.0005	0.030/1650	0.75	30	10
27	0.0005	0.030/1650	0.95	90	10
28	0.0005	0.050/1750	0.75	90	30
29	0.0005	0.050/1750	0.75	90	10
30	0.0005	0.030/1650	0.75	90	30
31	0.0005	0.050/1750	0.75	30	50
32	0.0005	0.050/1750	0.95	30	10
33	0.0005	0.030/1650	0.95	30	30
34	0.0005	0.050/1750	0.95	90	50
35	0.0005	0.050/1750	0.95	30	50
36	0.0005	0.030/1650	0.95	90	30
37	0.0005	0.050/1750	0.95	90	10
38	0.0005	0.050/1750	0.75	30	10
39	0.0005	0.030/1650	0.75	90	10
40	0.0005	0.050/1750	0.95	90	30
41	0.0005	0.030/1650	0.75	30	50
42	0.0005	0.030/1650	0.95	90	50
43	0.0005	0.030/1650	0.95	30	10
44	0.0005	0.030/1650	0.75	90	50
45	0.0005	0.030/1650	0.95	30	50
46	0.0005	0.050/1750	0.75	90	50
47	0.0005	0.050/1750	0.75	30	30
48	0.0005	0.030/1650	0.75	30	30

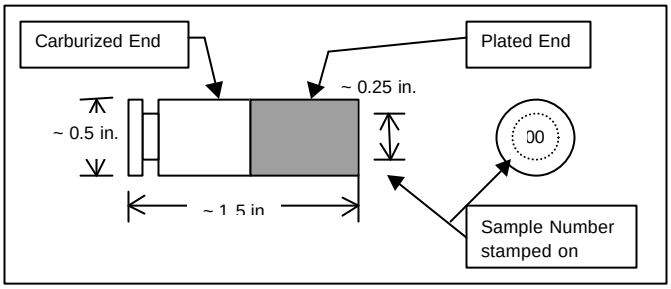


Figure 7 – Geometry of DOE 2 samples. In DOE 2, each of the samples was plated over only half its surface area, as indicated in the figure, according to the test matrix in Table 8.

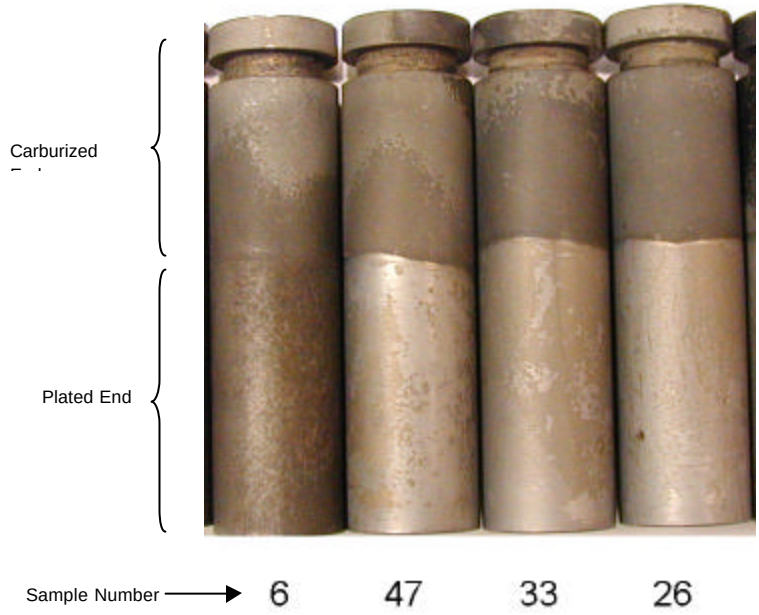


Figure 8 – Representative macroetched DOE 2 samples. Notice the sharp delineation in color at the interface between the plated and the carburized sections of each sample. Samples 6, 33 and 26 were assigned failing scores while sample 47 was given a passing score. The difficulty in distinguishing between samples 47, 33 and 26 is evidence of the subjectivity of the test.

Experimental Procedure

The parts were heat treated according to the following schedule, with randomization at appropriate intervals:

1) Carburize (According to the test matrix in Table 8)
2) Slow Cool
3) Subcritical Anneal at 1175°F in Air
4) Austenitize at 1525°F
5) Quench in Oil heated to 120°F
6) Cold Treat at -120°F
7) Temper at 300°F

The press quenching operation usually performed on selectively carburized parts was not performed in this experiment. A free-quench was determined as adequate.

Data and Analyses

A macroetch was performed, followed by microhardness analysis of the carburized case. The statistical examination used can be applied to almost any experiment. See [BHH78, Mon97] for a thorough review of the techniques used.

Macroetch Analysis

A macroetch was performed on the DOE 2 samples in a manner identical to that performed on the DOE 1 samples. Some samples were found to have evidence of carbon penetration upon inspection. In this case, each of the samples was given a qualitative pass/fail score to describe their condition. A score of “pass” was only given to specimens showing no evidence of penetration whatsoever. A failing score was given to samples showing the slightest sign of carbon penetration. In this sense, this test was very discriminative. But, one must keep in mind that it was also very subjective. The results are tabulated in Table 9. Failing scores in this test correlated to the higher carburizing temperature and the thinner plating thickness. The influence of carbon potential during austenitizing, austenitizing time and quench transfer time was found to be minimal based on these data.

Figure 8 is a photograph of a representative set of the macroetched DOE 2 samples.

Table 9 - Results of the DOE 2 macroetch. A failing score indicates that there was evidence of carbon penetration on the sample.

Sample Number	Score	Sample Number	Score	Sample Number	Score
1	Fail	17	Fail	33	Fail
2	Fail	18	Fail	34	Pass
3	Fail	19	Fail	35	Pass
4	Fail	20	Fail	36	Fail
5	Fail	21	Fail	37	Pass
6	Fail	22	Fail	38	Pass
7	Fail	23	Fail	39	Fail
8	Fail	24	Fail	40	Fail
9	Fail	25	Fail	41	Fail
10	Fail	26	Fail	42	Pass
11	Fail	27	Fail	43	Pass
12	Fail	28	Pass	44	Fail
13	Fail	29	Fail	45	Fail
14	Fail	30	Fail	46	Pass
15	Fail	31	Pass	47	Pass
16	Fail	32	Pass	48	Fail

Table 10 - Averaged hardness readings on DOE 2 samples at indicated intervals of the case depth. d represents depth [in]. At least three observations were made in each interval. Reported values are in HRC converted from Knoop according to ASTM E140. (continued on next page)

Sample Number	Plated end d = 0.002 in	Carburized end d = 0.002[4]	Carburized end 0.002 < d = 0.007
2	55.0	60.0	57.1
3	63.8	59.0	58.9
4	56.8	65.2	65.6
5	62.5	64.6	63.7
6	57.4	60.7	61.1

[4] One reading was taken at 0.0018 inch.

8	55.9	64.0	65.1
9	57.5	60.6	59.9

Table 10 - Averaged readings on DOE 2 samples at indicated intervals of the case depth. d represents depth [in]. At least three observations were made in each interval. Reported values are in HRC converted from Knoop according to ASTM E140. (concluded)

Sample Number	Plated end d = 0.002 in	Carburized end d = 0.002[5]	Carburized end 0.002 < d = 0.007
12	60.9	62.9	62.4
13	55.6	60.6	60.2
14	55.0	60.7	61.1
17	63.5	59.7	63.9
18	60.6	60.9	61.1
19	61.6	63.3	63.4
21	59.5	61.8	59.6
23	60.8	63.8	65.7
24	60.0	62.7	64.0
26	37.7	63.5	62.7
27	36.3	63.7	62.0
29	37.1	60.4	62.2
31	35.2	63.5	63.3
32	36.3	63.9	62.5
34	36.3	62.5	64.4
35	35.3	62.7	62.2
37	30.6	63.0	64.1
38	35.6	63.5	64.5
39	30.5	60.3	59.9
41	37.0	61.0	61.7
42	34.8	65.4	64.7
43	31.7	62.1	61.1
44	36.6	62.7	65.9

[5] One reading was taken at 0.0018 inch.

45	39.1	61.3	63.0
46	36.0	63.0	64.2

Microhardness Analysis

Microhardness data was gathered on 100% of the specimens. The samples were each polished to a 3 μm finish and the Knoop Hardness readings were taken using a Wilson Tukon Series 200 Microhardness Tester. A 500 g load, and 15 sec dwell time was used. Three microhardness traces were taken on each specimen for case analysis. Case depth was taken to be the depth into the sample surface at which the hardness dropped to 50 HRC. Note that the hardness was determined using the Knoop microhardness test (HK), but all values are reported in Rockwell C-scale hardness (HRC). All values reported in HRC are converted from Knoop microhardness using ASTM E140.

Average hardness values found in specified intervals of the case can be found in Table 10. Only the two extreme levels of the quench transfer time were used in this analysis.

The values in Table 10 were then run through MiniTab Release 12 [Min99]. MiniTab is a statistical package capable of producing factorial main-effects and interaction plots as well as performing analysis of variance (ANOVA). The main effects determined at each of the intervals in Table 10 are displayed in Figure 9. The main effects indicate that the most influential factor in the experiment was the copper plating thickness, with the 0.0001 in plating failing and the 0.0005 in plating performing well. The interaction plots also show that the factors examined in the experiment had little, if any effect on the carburized end of the specimen. Plating thickness played no role in the performance of the carburized end because it had no copper plating on it.

Figure 10 displays a graph of the standardized effects from the plated end of the specimen. This graph shows the relative influence of each of the factors based on the measured response, hardness at a depth of 0.002 inch This chart indicates that the most influential factor correlating to increasing hardness (failure) was the copper plating thickness.

ANOVA performed on the data yield a high significance for the copper plating thickness playing the most influential role in failure of the plating. The complete fully nested ANOVA generated by MiniTab is included in Appendix D. The next most influential effect was the interaction of copper plating thickness and the time spent in the rotary furnace: The measured value of microhardness increased with thinner plating and longer time spent in the rotary furnace. This effect was, however, negligible compared to the effect of copper plating thickness alone. Figure 11 displays this secondary interaction plot and shows this relationship.

The microhardness data collected was plotted to gain a qualitative understanding of the response of the material to the levels of the factors examined in the experiment. Figure 12 displays the graphs generated. Figure 12(a) displays the data obtained in the DOE 2 microhardness analysis, split across the copper plating levels in the test matrix. Curves were fit to the data to gain a qualitative understanding of the results. The trendlines on each graph was subjectively fit using regression routines in Microsoft Excel. Trendlines for the data obtained from the carburized ends (designated CE on the graphs) of the specimens were fit using polynomials. Similarly, trendlines for the data obtained from the plated ends of

the specimens were fit using the power equation, $y = cx^b$. The only exception to this was the data for the plated end (0.0001 inch) in Figure 12(a), which was fit using a polynomial.

Figure 12(a) shows that the plated end of the specimens plated to 0.0001 inch were carburized to a case depth of 0.006 inch The trendline for the data obtained from the plated end of the specimens plated to 0.0005 inch shows a downward trend near the surface. This was determined to be due to data that was taken too close to the surface (0.002 inch) of the "soft" end. Notice the wide spread in the data obtained near the surface and the subsequent "thinning" of the spread as depth increases.

Figure 12(b), a similar correlation graph for the effect of carburizing potential in the austenitizing furnace (rotary furnace). This graph shows no substantial correlation between either of the levels, 0.75% and 0.95%.

Figure 12(c) shows trendlines for the data split across the different carburizing levels. The case depths achieved in the experimentation were 0.026 inch and 0.039 inch, compared to the 0.030 inch and 0.050 inch in the test matrix. The graph shows no correlation between the carburizing temperature / case depth on the plated end.

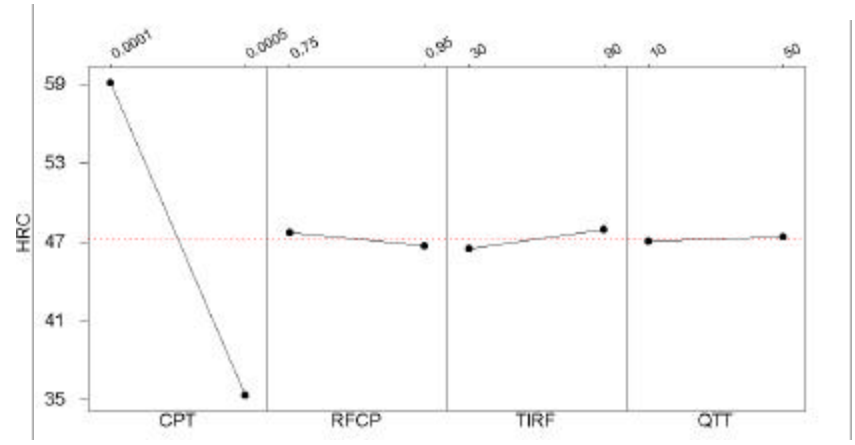
Figure 12(d) shows the effect of time spent in the austenitizing furnace on the plated end of the specimens. It can be seen from the graph that longer time spent in the rotary furnace leads to greater carburizing penetration, as one would expect from Fick's diffusion relations [Gli00, KY87].

In Figure 12(e), the data is split across the quench transfer times tested in the experiment. No differences are evident in the trendlines fit to the data, meaning that the time it takes for the part to leave the protective atmosphere of the austenitizing furnace can vary between 10 and 50 sec. (inclusive) with no detrimental affects to the hardness of the part surface.

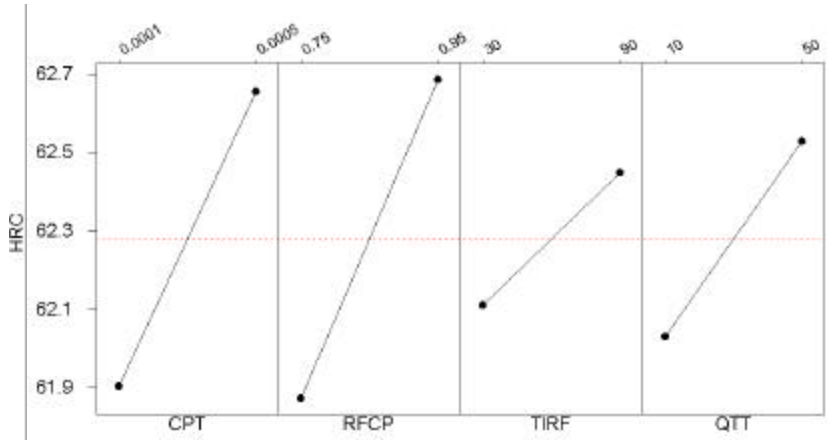
DOE 2 Conclusions

The main conclusion from DOE 2 was that the 0.0001 inch copper plating failed in an SAE/AISI 9310H heat treatment cycle, while 0.0005 inch plating performed as required. Secondary conclusions were that the factors of rotary furnace carbon potential, time in the rotary furnace, and quench transfer time did not play a significant role in the performance of the copper plating. Neither did these factors play a significant role in the response measured on the carburized end of each specimen, as evidenced by the Pareto chart in Figure 10. From those conclusions, it can be stated that (assuming all the relationships are linear, as assumed in the analysis) the variables examined could be set anywhere within the bounds of the levels examined and yield adequate performance.

The parts were visually inspected after each major operation during the experimentation. Of note is the inspection following the subcritical-anneal. Visual inspection following the subcritical-anneal step revealed evidence of spalling of the copper plating. It was conjectured that this may be due to the air atmosphere that the anneal is performed under. Because this particular operation is not always performed on parts, and because of the evidence of spalling at this process point, this was chosen as a factor to examine in DOE 3.

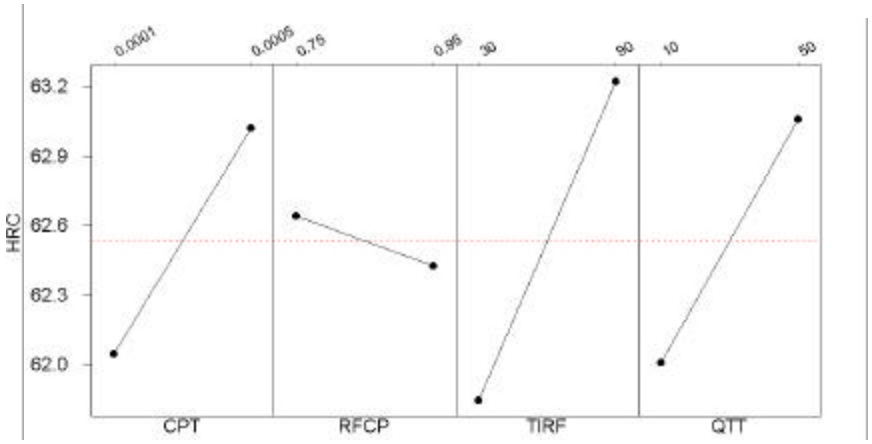


(a)



(b)

Figure 9 - Main effects plots for each of the intervals of values reported in Table 10. Factors plotted (from left to right) are: copper plating thickness (CPT), rotary furnace carbon potential (RFCP), austenitizing time (TIRF), and quench transfer time (QTT). (a) Plated end at $d \leq 0.002$. (b) Carburized end at $d \leq 0.002$. (c) Carburized end at $0.002 < d \leq 0.007$. Notice the difference in the ordinate scales between the three plots. (continued on next page)



(c)

Figure 9 - Main effects plots for each of the intervals of values reported in Table 10. Factors plotted (from left to right) are: copper plating thickness (CPT), rotary furnace carbon potential (RFCP), austenitizing time (TIRF), and quench transfer time (QTT). (a) Plated end at d = 0.002. (b) Carburized end at d = 0.002. (c) Carburized end at 0.002 < d = 0.007. Notice the difference in the ordinate scales between the three plots. (concluded)

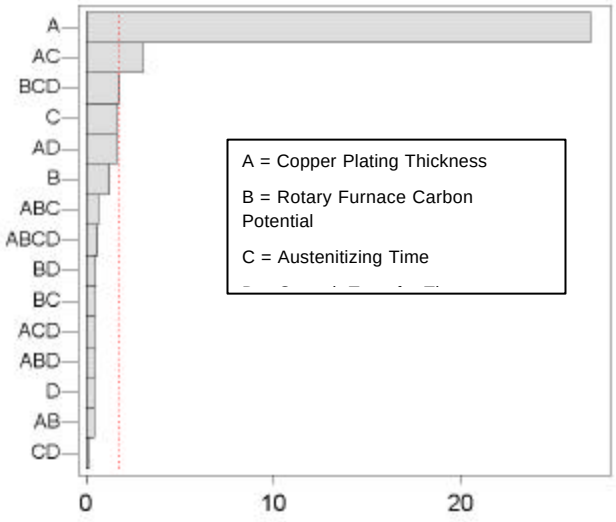


Figure 10 – Pareto chart of the standardized effects in DOE 2 based on microhardness data taken at a depth of 0.002 inch from the copper plated end of the specimen. This chart indicates that factor A, copper plating thickness, was the most influential in failure of the plating.

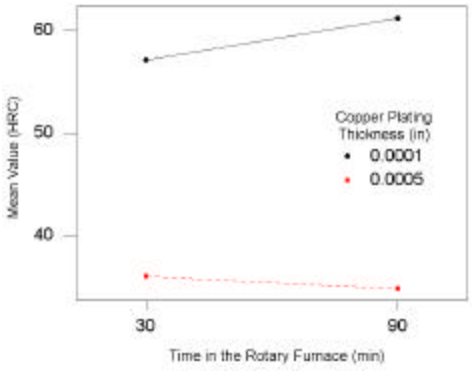
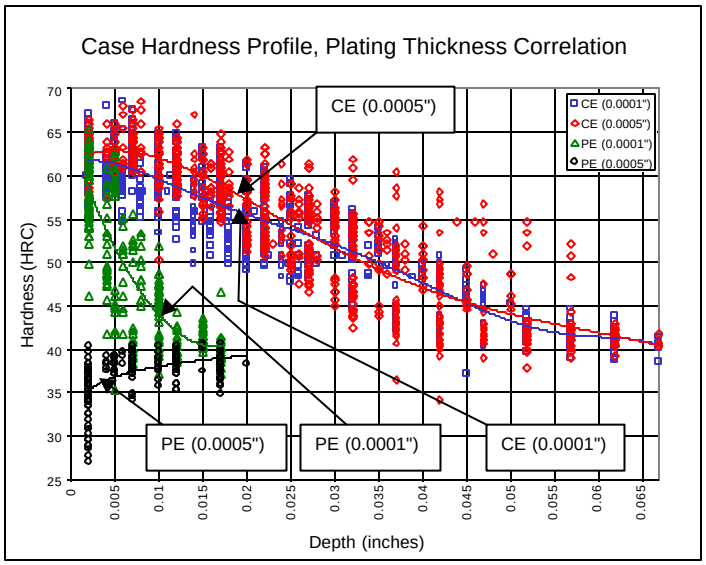
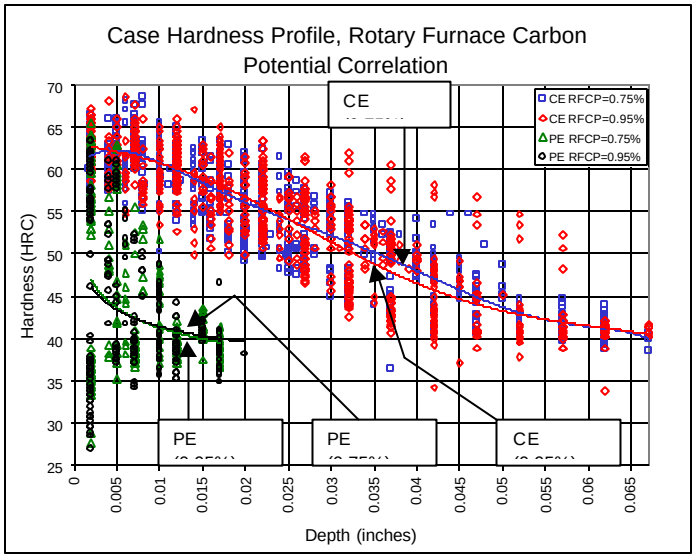


Figure 11 – Secondary Effects Plot highlighting the correlation between copper plating thickness and time spent in the rotary furnace. The measured value of microhardness increased with thinner plating and longer time spent in the rotary furnace, as evidenced by the diverging gap between the two curves.

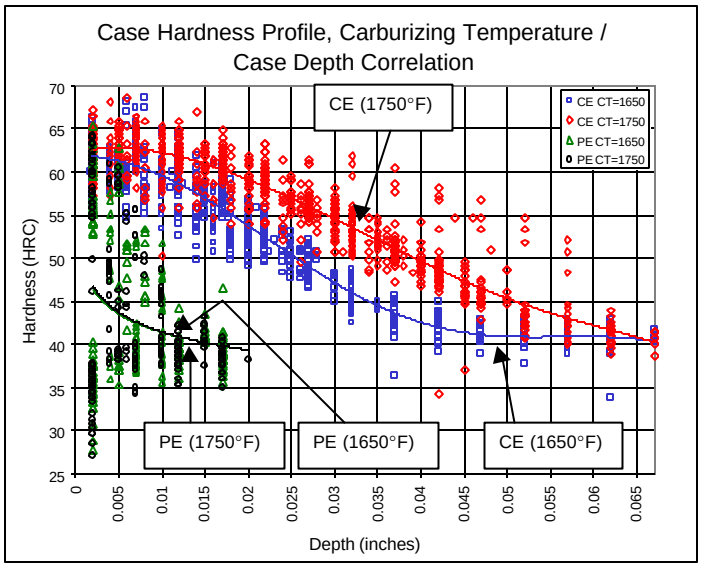


(a)

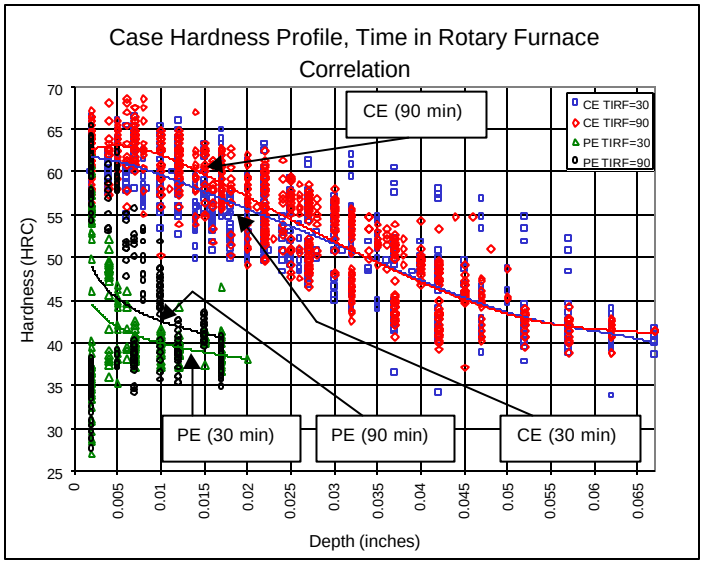


(b)

Figure 12 – Graphs of the DOE 2 microhardness data. These plots illustrate a qualitative analysis of the main effects in the DOE. All values are in HRC, converted from HK using ASTM E140. CE refers to the carburized-end, PE to the plated-end. (continued on next page)

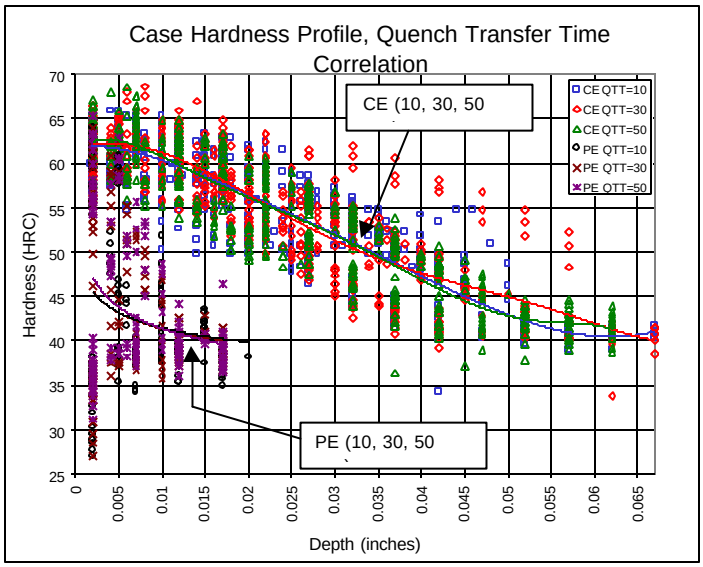


(c)



(d)

Figure 12 – Graphs of the DOE 2 microhardness data. These plots illustrate a qualitative analysis of the main effects in the DOE. All values are in HRC, converted from HK using ASTM E140. CE refers to the carburized-end, PE to the plated-end. (continued on next page)



(e)

Figure 12 – Graphs of the DOE 2 microhardness data. These plots illustrate a qualitative analysis of the main effects in the DOE. All values are in HRC, converted from HK using ASTM E140. CE refers to the carburized-end, PE to the plated-end. (concluded)

DOE 3

Overview

The purpose of DOE 3 was to verify the findings in DOE 2 and to expand the experimentation to new factors and levels. The goal was to confirm the findings of DOE 2.

Once again, discussion between IITRI and Honeywell personnel led to the following factors/levels being examined.

1) Copper Plating Thickness
   a) 0.0003 inch
   b) 0.0005 inch
2) Carburizing Temperature
   a) 1650°F, 0.030 inch case depth
   b) 1700°F, 0.050 inch case depth
   c) 1750°F, 0.070 inch case depth
3) Subcritical Anneal
   a) 1175°F
   b) 80°F (Room temperature)
4) Rotary Furnace Carbon Potential
   a) 0.65%
   b) 0.85%

Because the 0.0001 inch plating failed in the last experiment, while the 0.0005 inch plating performed well, an intermediate plating thickness of 0.0003 in was chosen.inch Once again, carburizing temperature was used as a blocking variable and three temperature/case depth combinations were chosen. Honeywell does not perform a subcritical anneal on their parts, however other heat treaters in the industry do, so this time the subcritical anneal was chosen as a factor to examine. The absence of a subcritical anneal for half the factorial expansion was chosen as a “room temperature treatment” for purposes of the quantitative analysis, using MiniTab. Carbon potential in the rotary furnace was chosen as a factor again, this time to lower the level and see if a “bottom” could be found in the factor.

Additionally, a duplicate sample set was added to evaluate the protective effect of copper plating in the rotary furnace after the samples had been carburized. In effect, this second sample set (samples 49-96) isolated the effect of the rotary furnace.

The DOE 3 test matrix can be found in Tables 11 (samples plated prior to carburizing) and 12 (samples plated after carburizing). The sample geometry changed substantially in this

experiment, going from the bars in DOEs 1 and 2 to a small gear. This new sample geometry can be found in Figure 13.

Table 11 – DOE 3 test matrix. Samples plated prior to carburizing. (continued on next page)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Subcritical Annealing Temperature (°F)	Rotary Furnace Carbon Potential (%)
1	0.0003	0.030 / 1650	1175	0.85
2	0.0003	0.070 / 1750	1175	0.85
3	0.0003	0.030 / 1650	80	0.65
4	0.0003	0.050 / 1700	1175	0.65
5	0.0003	0.070 / 1750	80	0.65
6	0.0003	0.050 / 1700	80	0.85
7	0.0003	0.030 / 1650	1175	0.65
8	0.0003	0.070 / 1750	1175	0.65
9	0.0003	0.050 / 1700	80	0.65
10	0.0003	0.070 / 1750	80	0.85
11	0.0003	0.030 / 1650	80	0.85
12	0.0003	0.050 / 1700	1175	0.85
13	0.0003	0.030 / 1650	1175	0.85
14	0.0003	0.050 / 1700	1175	0.65
15	0.0003	0.070 / 1750	80	0.85
16	0.0003	0.030 / 1650	80	0.65
17	0.0003	0.050 / 1700	80	0.65
18	0.0003	0.050 / 1700	80	0.85
19	0.0003	0.030 / 1650	80	0.85
20	0.0003	0.070 / 1750	1175	0.65
21	0.0003	0.070 / 1750	80	0.65
22	0.0003	0.030 / 1650	1175	0.65
23	0.0003	0.070 / 1750	1175	0.85
24	0.0003	0.050 / 1700	1175	0.85

Table 11 – DOE 3 test matrix. Samples plated prior to carburizing. (concluded)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Subcritical Annealing Temperature (°F)	Rotary Furnace Carbon Potential (%)
25	0.0005	0.030 / 1650	80	0.65
26	0.0005	0.070 / 1750	80	0.85
27	0.0005	0.050 / 1700	80	0.65
28	0.0005	0.030 / 1650	1175	0.85
29	0.0005	0.070 / 1750	1175	0.65
30	0.0005	0.070 / 1750	1175	0.85
31	0.0005	0.030 / 1650	80	0.85
32	0.0005	0.050 / 1700	1175	0.85
33	0.0005	0.050 / 1700	1175	0.65
34	0.0005	0.030 / 1650	1175	0.65
35	0.0005	0.070 / 1750	80	0.65
36	0.0005	0.050 / 1700	80	0.85
37	0.0005	0.050 / 1700	1175	0.65
38	0.0005	0.050 / 1700	80	0.65
39	0.0005	0.030 / 1650	80	0.85
40	0.0005	0.070 / 1750	80	0.65
41	0.0005	0.070 / 1750	1175	0.85
42	0.0005	0.030 / 1650	1175	0.65
43	0.0005	0.070 / 1750	80	0.85
44	0.0005	0.050 / 1700	80	0.85
45	0.0005	0.030 / 1650	1175	0.85
46	0.0005	0.050 / 1700	1175	0.85
47	0.0005	0.030 / 1650	80	0.65
48	0.0005	0.070 / 1750	1175	0.65

Table 12 – DOE 3 test matrix. Samples plated after carburizing. (continued on next page)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Subcritical Annealing Temperature (°F)	Rotary Furnace Carbon Potential (%)
49	0.0003	0.05	80	0.85
50	0.0003	0.03	80	0.85
51	0.0003	0.07	80	0.65
52	0.0003	0.07	80	0.85
53	0.0003	0.03	1175	0.65
54	0.0003	0.03	1175	0.85
55	0.0003	0.05	1175	0.85
56	0.0003	0.07	1175	0.65
57	0.0003	0.03	80	0.65
58	0.0003	0.05	80	0.65
59	0.0003	0.07	1175	0.85
60	0.0003	0.05	1175	0.65
61	0.0003	0.07	1175	0.85
62	0.0003	0.05	1175	0.65
63	0.0003	0.03	80	0.85
64	0.0003	0.05	80	0.65
65	0.0003	0.07	80	0.65
66	0.0003	0.03	1175	0.65
67	0.0003	0.07	80	0.85
68	0.0003	0.07	1175	0.65
69	0.0003	0.03	80	0.65
70	0.0003	0.05	1175	0.85
71	0.0003	0.05	80	0.85
72	0.0003	0.03	1175	0.85

Table 12 – DOE 3 test matrix. Samples plated after carburizing. (continued on next page)

Sample Number	Copper Plating Thickness (in)	Case Depth (in) / Temperature (°F)	Subcritical Annealing Temperature (°F)	Rotary Furnace Carbon Potential (%)
73	0.0005	0.05	80	0.65
74	0.0005	0.07	80	0.85
75	0.0005	0.03	80	0.65
76	0.0005	0.05	80	0.85
77	0.0005	0.03	1175	0.65
78	0.0005	0.07	1175	0.85
79	0.0005	0.05	1175	0.65
80	0.0005	0.03	1175	0.85
81	0.0005	0.07	80	0.65
82	0.0005	0.07	1175	0.65
83	0.0005	0.03	80	0.85
84	0.0005	0.05	1175	0.85
85	0.0005	0.07	80	0.85
86	0.0005	0.03	80	0.65
87	0.0005	0.05	1175	0.65
88	0.0005	0.05	80	0.65
89	0.0005	0.03	80	0.85
90	0.0005	0.07	80	0.65
91	0.0005	0.05	1175	0.85
92	0.0005	0.03	1175	0.65
93	0.0005	0.05	80	0.85
94	0.0005	0.07	1175	0.65
95	0.0005	0.03	1175	0.85
96	0.0005	0.07	1175	0.85

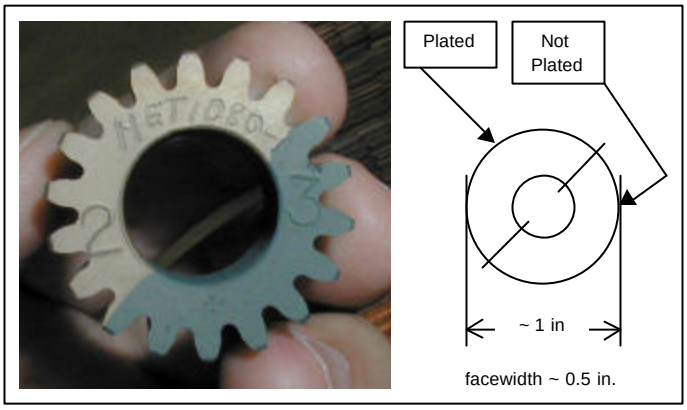


Figure 13 – Geometry of DOE 3 samples. The samples processed in DOE 3 were of a substantially different geometry than DOEs 1 and 2 (see Figure 5 or 7 to compare). In DOE 3, each sample was plated over only half its surface area, as indicated in the figure, according to the test matrix in Tables 11 and 12. This particular picture of sample 23 was taken after carburization.

Experimental Procedure

DOE 3 experimentation was almost identical to that of DOE 2. Quench transfer time was not a factor in this experiment, but was controlled between 10 and 50 sec.

The parts were heat treated according to the following schedule, with randomization at appropriate intervals:

1) Carburize (According to the test matrix in Tables 11 and 12)
2) Slow Cool
3) Subcritical Anneal at 1175°F in Air (According to the test matrix in Tables 11 and 12)
4) Austenitize at 1525°F
5) Quench in Oil heated to 120°F
6) Cold Treat at -120°F
7) Temper at 300°F

As with the previous experiment, a free-quench rather than press quench was performed on the samples.

Two surfaces were available for examination on each specimen, similar to the specimens in DOE 2.

Macroetch Analysis

A macroetch was performed on the DOE 3 samples in a manner identical to that performed on the DOE 1 and 2 samples. Again, this was a very discriminatory inspection; any sample with signs of possible leakage was given a failing score. The results are presented in Table 13. The macroetch indicated that samples that were subjected to a subcritical anneal were the most negatively affected, confirming the suspicions from DOE 2.

Microhardness Analysis

As with DOE 2, microhardness data was gathered on 100% of the specimens. Again, the samples were each polished to a 3 μm finish and the Knoop Hardness readings were taken using a Wilson Tukon Series 200 Microhardness Tester. And again, a 500 g load, and 15 sec dwell time was used. This time, measurements were only taken at a depth of 0.003 inch Three measurements were made on each surface. The measurements were taken on the inside diameter of the gear specimens to avoid 2-dimensional effects that would have been encountered on the outer diameter of the specimens, due to the 2-dimensional tooth geometry. Individual measurements can be found in Table 14.

Three major analyses were performed on this response using MiniTab:

A) ANOVA of the plated halves of sample numbers 1 through 48

B) ANOVA of the plated halves of sample numbers 49 through 96

C) ANOVA of the unplated halves of sample numbers 1 through 96

Appendix E contains a fully nested ANOVA for the analysis performed on the plated halves of the samples plated prior to carburizing. Based on the ANOVA, the subcritical anneal and the interaction of the case depth and subcritical anneal were the only significant effects observed. Figure 14(a) contains the main effects for this ANOVA. Figure 15 displays the secondary effects plot for the interaction of the case depth and subcritical anneal. The

Appendix F contains a fully nested ANOVA for the analysis performed on the plated halves of the samples plated after carburizing. Figure 14(b) contains the main effects for this ANOVA. All effects in this analysis were hidden by error.

Appendix G contains a fully nested ANOVA for the analysis performed on the unplated halves of all the samples in DOE 3. Figure 14(c) contains the main effects for this ANOVA. The major difference separating this analysis from the others in this DOE was that all the samples were investigated. The major difference between the two samples sets was that one set (samples 1 through 48) was treated to a plating process before carburization. The other set (samples 49 through 96) was treated to a plating process after carburization. This

was treated as a factor in the analysis and levels of –1 and 1 were assigned. Similar to the ANOVA in Appendix F, all effects in this analysis were hidden by error.

DOE 3 Conclusions

Based on analysis A, the ANOVA of the plated halves of the sample numbers 1 through 48 and its accompanying main effects, secondary effects and pareto plots, it can be said that the subcritical anneal at 1175°F was the most detrimental factor influencing failure of the plating. The macroetch performed on the samples supports this conclusion.

Based on this data, the macroetch, a much simpler analysis to perform than the microhardness analysis, can be used as a simple qualitative and quick tool to gauge failure of the plating. However, the subjectivity of the test will probably disallow its use in production.

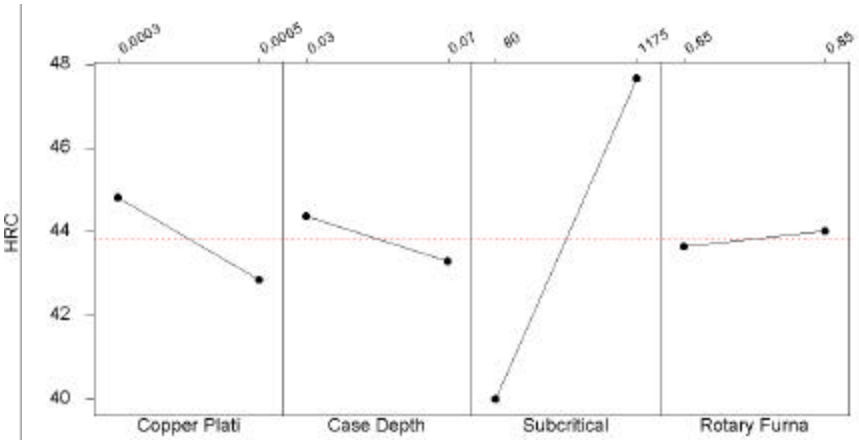
The analysis performed on the plated-after-carburization specimens indicated that any effects present were hidden by error. All main effects were within 1.5 HRC of each other, well within the error expected when examining hardness using the Knoop method (see Appendix X1 and X2 of ASTM E384).

The analysis performed on the non-plated halves of the specimens indicated that all main effects were within 0.5 HRC, again hidden by error in the measurements.

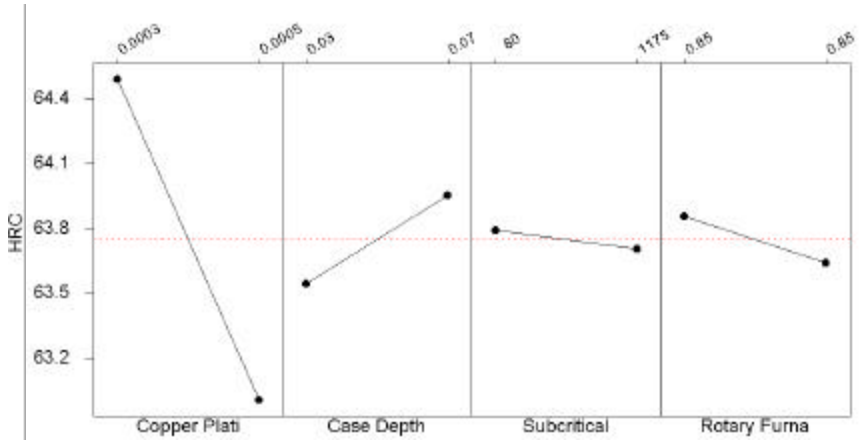
Table 13 - Results of the DOE 3 macroetch. A failing score indicates that there was evidence of carbon penetration on the sample.

Sample Number	Score	Sample Number	Score	Sample Number	Score
1	Fail	33	Fail	65	Fail
2	Pass	34	Fail	66	Fail
3	Pass	35	Pass	67	Fail
4	Fail	36	Pass	68	Fail
5	Pass	37	Fail	69	Fail
6	Pass	38	Pass	70	Fail
7	Fail	39	Pass	71	Fail
8	Fail	40	Fail	72	Fail
9	Pass	41	Fail	73	Fail
10	Pass	42	Fail	74	Fail
11	Pass	43	Pass	75	Fail
12	Fail	44	Pass	76	Fail
13	Fail	45	Fail	77	Fail
14	Fail	46	Fail	78	Fail
15	Pass	47	Pass	79	Fail
16	Pass	48	Pass	80	Fail
17	Pass	49	Fail	81	Fail
18	Pass	50	Fail	82	Fail
19	Pass	51	Fail	83	Fail
20	Fail	52	Fail	84	Fail
21	Pass	53	Fail	85	Fail
22	Fail	54	Fail	86	Fail
23	Fail	55	Fail	87	Fail
24	Fail	56	Fail	88	Fail
25	Fail	57	Fail	89	Fail
26	Pass	58	Fail	90	Fail
27	Pass	59	Fail	91	Fail
28	Fail	60	Fail	92	Fail
29	Fail	61	Fail	93	Fail
30	Fail	62	Fail	94	Fail

31	Pass	63	Fail	95	Fail
32	Fail	64	Fail	96	Fail

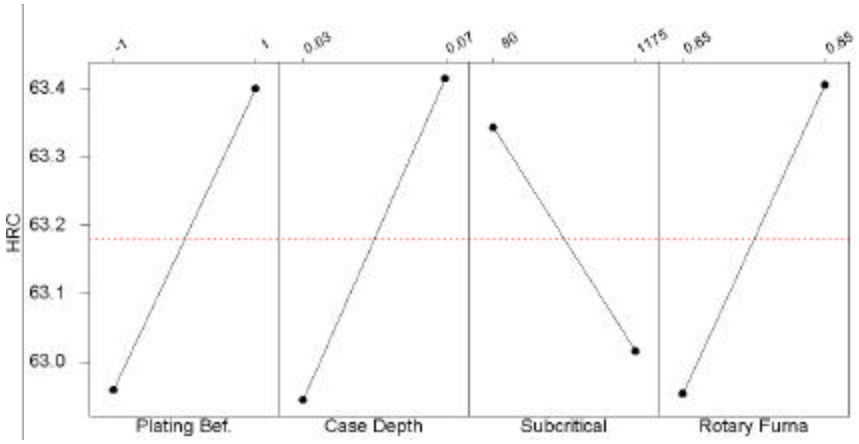


(a)



(b)

Figure 14 – Main effects plots for DOE 3. Factors plotted (from left to right) are: copper plating thickness, case depth, subcritical annealing temperature, and rotary furnace carbon potential. (a) Main effects for the plated halves of the samples plated prior to carburizing. (b) Main effects for the plated halves of the samples plated after carburizing. (c) Main effects for the unplated halves of all the samples in DOE 3. In (c), the two sample sets used for the previous two analyses were combined and treated as a factor with two levels (-1, 1); one sample set treated to a plating operation prior to carburization, the other after. Notice the differences in the ordinate scales between the three plots. (continued on next page)



(c)

Figure 14 – Main effects plots for DOE 3. Factors plotted (from left to right) are: copper plating thickness, case depth, subcritical annealing temperature, and rotary furnace carbon potential. (a) Main effects for the plated halves of the samples plated prior to carburizing. (b) Main effects for the plated halves of the samples plated after carburizing. (c) Main effects for the unplated halves of all the samples in DOE 3. In (c), the two sample sets used for the previous two analyses were combined and treated as a factor with two levels (-1, 1); one sample set treated to a plating operation prior to carburization, the other after. Notice the differences in the ordinate scales between the three plots. (continued on next page)

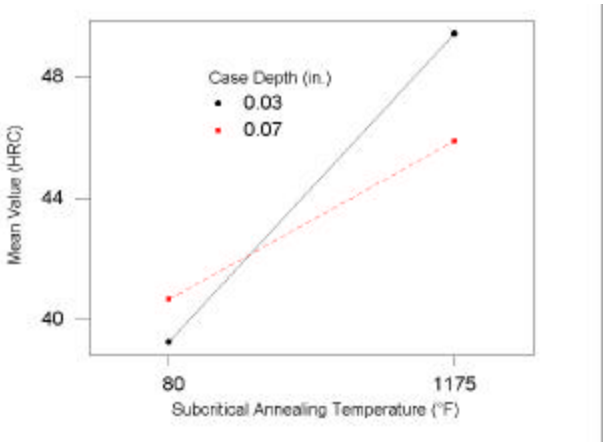


Figure 15 – Secondary effects plot for DOE 3 analysis A, the interaction of the case depth and subcritical anneal. This effects was minimal compared to the main effect, see Figure 16.

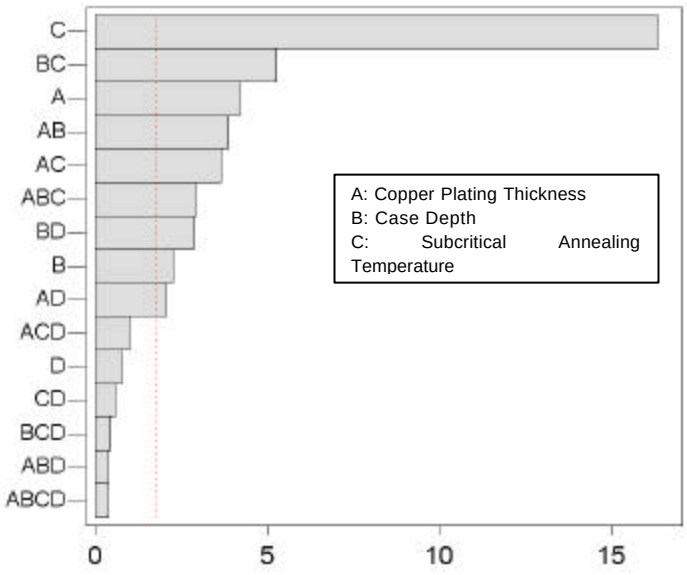


Figure 16 – Pareto chart of the standardized effects in DOE 3 based on microhardness data taken at a depth of 0.003 inch This chart indicates that factor C, subcritical annealing temperature, was the most influential in failure of the plating. The interaction of factors B and C were the second most influential, but was only one third as powerful as the main effect.

Table 14 – Hardness readings from DOE 3 samples on indicated half. All readings were taken at 0.003 in below the surface. Reported values are in HRC converted from Knoop according to ASTM E140. (continued on next page)

Samples Number	Reading From Plated Half (HRC)	Reading From Carburized Half (HRC)	Samples Number	Reading From Plated Half (HRC)	Reading From Carburized Half (HRC)
1	53.0	65.4	25	39.2	65.4
2	48.0	63.9	26	40.0	65.9
3	37.1	61.4	27	39.6	65.4
4	45.0	65.4	28	49.1	62.8
5	41.0	64.6	29	46.1	63.3
6	40.7	62.8	30	41.7	64.4
7	48.3	61.4	31	39.6	57.8
8	48.0	62.8	32	47.6	62.8
9	41.0	63.9	33	46.5	64.4
10	40.3	63.9	34	48.7	62.8
11	39.6	62.4	35	41.0	64.9
12	48.3	62.8	36	39.0	65.9
13	49.5	64.9	37	45.0	63.9
14	45.7	61.9	38	38.2	60.0
15	41.4	65.9	39	40.0	63.9
16	38.6	64.9	40	40.3	63.3
17	41.7	65.9	41	40.3	62.6
18	43.6	64.9	42	49.5	62.8
19	41.4	63.9	43	40.0	62.4
20	50.7	54.2	44	41.7	63.3
21	41.4	62.8	45	49.5	61.4
22	48.0	63.3	46	53.6	63.9
23	50.7	63.9	47	38.6	61.9
24	49.4	64.9	48	41.7	59.5

Table 14 – Readings from DOE 3 samples on indicated half. All readings were taken at 0.003 in below the surface. Reported values are in HRC converted from Knoop according to ASTM E140. (concluded)

Samples Number	Reading From Plated Half (HRC)	Reading From Carburized Half (HRC)	Samples Number	Reading From Plated Half (HRC)	Reading From Carburized Half (HRC)
49	63.3	63.9	73	62.4	62.4
50	64.9	63.3	74	64.4	61.4
51	64.9	62.8	75	63.3	61.9
52	62.4	62.9	76	57.7	60.0
53	64.4	63.9	77	61.4	61.9
54	64.9	62.8	78	57.7	63.3
55	63.3	62.8	79	61.9	60.9
56	65.9	66.4	80	61.4	61.9
57	63.3	65.4	81	63.9	61.9
58	64.4	64.4	82	64.4	61.3
59	63.9	64.9	83	60.5	61.9
60	64.9	63.9	84	61.4	60.0
61	65.4	64.3	85	63.3	63.9
62	60.0	62.4	86	62.8	61.9
63	65.4	63.9	87	61.4	60.9
64	65.5	64.4	88	60.0	59.5
65	65.4	64.9	89	63.9	62.4
66	64.9	63.9	90	62.4	62.4
67	65.5	65.9	91	60.9	61.4
68	62.4	63.3	92	62.4	61.9
69	64.4	64.9	93	63.3	61.9
70	64.4	62.3	94	65.5	66.5
71	63.3	64.9	95	64.9	65.4
72	63.9	60.5	96	65.9	64.9

Diffusion Model

Purpose and Goal

The purpose of modeling the behavior of the copper/carbon system was to try to gain an understanding of the mechanisms at work that would likely cause the copper diffusion barrier to fail. Much work has been done on the diffusion of carbon in copper for its application in composite systems [DF95, DF96, DF97, DF98. DFS99], but not with respect to application in selective carburizing.

Introduction

Carbon is almost completely insoluble in copper (T_m = 1982.34°F) up to very high temperatures. Solubility does not exceed 0.04 at.% [BAPD90]. Solubility at carburizing temperatures (1650 – 1750°F is approximately 0.02 at.% [BAPD90]. The copper lattice is FCC and carbon is most likely to occupy octahedral sites, rather than tetrahedral sites because the octahedral sites are approximately twice as large [DF95]. For comparison, ionic radii of the atoms are $R_{Cu} \approx 0.75$ Å and $R_C \approx 0.15$ Å [Lid00]. Octahedral sites possess a radius of $R_{OS} = 0.31$ Å based on R_{Cu}. The crystal structure of C (graphite) is HCP.

Solubility is modified by the addition of alloying elements in copper, but in general the solubility decreases [DF95].

Based on this, diffusion of carbon through copper and into the SAE/AISI 9310H substrate is unlikely. However, the copper diffusion barrier can still fail, as demonstrated in the preceding experimentation. Failure could be due to the following detrimental conditions:

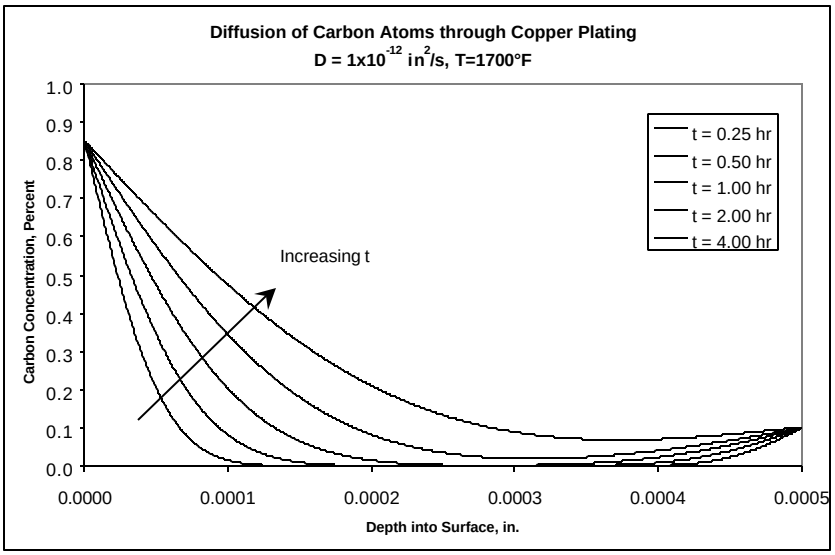
1) Porosity
2) Local reduction in layer thickness (possibly due to current density effects)
3) Mechanical damage such as nicks or scratches
4) Adherence problems
5) Chemical attack during adhesive removal operation
6) Oxidation of the layer at high temperature

Once carbon has entered the substrate through one of these defects, diffusion into the steel can proceed easily.

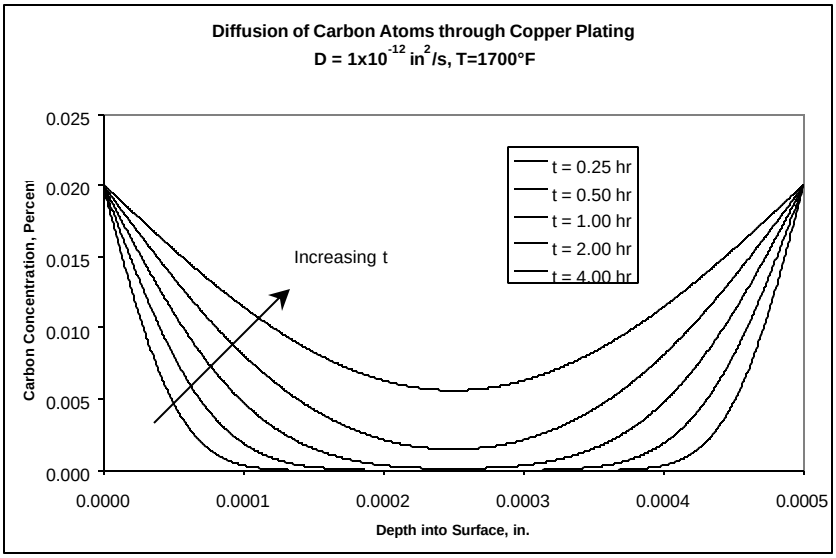
Methodology

The diffusion system was first modeled using an error function solution to Fick's second law. The first step was to model the system assuming infinite solubility of carbon in copper. The resulting curves can be found in Figure 17(a). Figure 17(b) shows what this system

looks like with the more realistic, limited, solubility mentioned earlier. Notice the different ordinate scales between Figures 17(a) and 17(b).



(a)



(b)

Figure 17 – Model of carbon diffusion through copper plating (0.0005 inch). Similar curves could be generated using different values of D (estimated) and t. (a) Assuming infinite

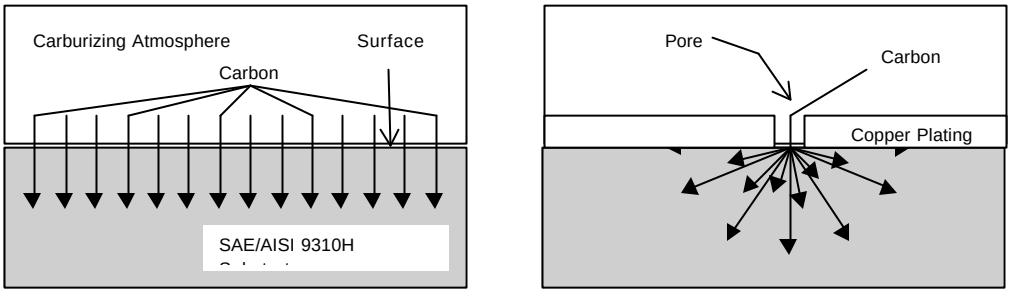
solubility. (b) Assuming limited solubility. Both (a) and (b) use the same values of D and t. Notice the different ordinate scales

The model in Figure 17 is a superposition of an error function solution to Fick's second law. It should be noted that this solution breaks down as soon as the left and right solutions begin to interact, however, the model serves to give the reader a qualitative understanding of the phenomena. This model breaks down at large values of $\sqrt{Dt}$ because the fields begin to interact at their boundaries. This model assumes ideal conditions such as: uniformity of plating thickness, no porosity, and no current density effects. A more rigorous approach would be to attempt to describe the system from the standpoint of all these flaws.

Conclusions

Because, as stated earlier, the system cannot easily diffuse carbon through the copper matrix, the diffusion must be entering the substrate via flaws. Voids (pores) are regularly present in copper plating. Diffusion in the region of these voids is three-dimensional, compared to the one-dimensional diffusion that occurs at exposed surfaces. This difference is illustrated in Figure 18. Two dimensional diffusion occurs in the volumes near edges on parts, but is of no concern here. Three dimensional diffusion of copper from a point source, such as a void, occurs slower than diffusion from a wall source, such as a surface.

Porosity in copper plating is commonly measured using the “ferroxyl" method (See the State of the Art Review in Appendix A). Manufacturers, as of this writing, monitor porosity on an as-needed basis for the conventional plating thicknesses of 0.001 inch to 0.003 inch. They did mention, during the industry survey (Again, Appendix A) that they would like to implement SPC techniques in there monitoring processes if possible. Thinner plating may need to be checked more often. Statistical analysis of porosity was not examined in this project.



(a) (b)

Figure 18 – Diffusion into a substrate. Bold arrows indicate carbon. (a) One-dimensional. (b) Three-dimensional.

Cost-Benefit Analysis

Introduction

The experiments conducted at IITRI prove that copper plating of thicknesses on the order of 0.0001 to 0.0005 inch are capable of stopping off carbon for the carburizing-only cycles tested. Manufacturers can take advantage of this data and use thinner plating thicknesses in their selective carburizing processes. This will result in a significantly shorter amount of time spent in plating baths, resulting in shorter cycle times, reduced labor and consequently reduced cost. Additionally, reduced cyanide usage and reduced copper anode usage can be expected from the reductions.

The experiments run by IITRI also prove that thinner-than-conventional copper thicknesses can be used throughout an entire heat treatment cycle. Plating thicknesses as low as 0.0001 and 0.0003 inch cannot be used, as is evident in the data from DOEs 2 and 3, however 0.0005 inch plating did not show signs of leakage on the specimens tested. The reader must keep in mind that the conclusions reached for this are only valid for the range of levels in the experiments performed. As such, performing a subcritical anneal in air has a high potential for failure of the thinner plating, and consequently the thinner plating cannot be used if production cycles similar to the ones tested require an unprotected subcritical annealing operation.

The Low Copper Project results have demonstrated that the present process of manufacturing precision gears can be changed to reduce the cost of the gear. This cost reduction is sufficient to offset the cost of the project in less than one year. The projected savings is based on the following assumptions:

1) The helicopter fleet consists of 4,250 rotary winged craft
2) Ten percent of the fleet will require spare sets of gears each year
3) Thirty percent of each helicopter gear set will benefit from the modified manufacturing process

The number of gears in each set is identified in Table 15.

The conventional process for the manufacture of precision gears is to clean the gear surface and selectively copper plate the gears to a thickness of 0.001 - 0.003 inch. After plating, the gears are carburized, slow cooled, and subcritical annealed. The copper plating is chemically stripped from the steel and then re-plated to a thickness of 0.0005 inch. The parts are then reheated in a furnace, austenitized, and press quenched. After the press quenching operation, the parts are cold treated, tempered, and then cleaned. When the cleaning is completed, the copper plating is chemically stripped and the part is inspected.

Table 15 – The U.S. Army helicopter fleet and their respective gear sets (year 2000 numbers). Gear-set numbers were calculated from tear-down manuals.

Helicopter Type	Year 2000 qty.	Gear Set Qty.
AH-1 Cobra	250	68
UH-1 Iroquois	685	60
OH-58C/D Kiowa Warrior	495	60
CH-47D Chinook	445	39
UH-60 A/6 Blackhawk	1500	34
AH-64 A/D Apache Longbow	750	68
Miscellaneous Helicopters	125	34
Total	4250	52 (Average)

The process of copper plating to a thickness of 0.001 inch to 0.003 inch to mask areas where carburization is not desired can be modified. The process modification would be the reduction of copper plating to a thickness of 0.0005 inch, which would provide satisfactory masking against carburization. The benefit of this process change is that less time is spent in the plating tank, and a cost reduction is achieved.

Subsequently, the gear would be heated in an austenitizing furnace, press quenched, cold treated, tempered, stripped and cleaned. Lastly, the copper would be removed by chemically stripping the 0.0005 inch copper off the surface.

The new process eliminates the 0.001 inch to 0.003 inch copper plating operation. Additionally, the resulting reduced copper plating thickness used eliminates the need to strip the thicker copper plating from the steel prior to the press quench operation. This elimination of the thicker copper plating operation and the stripping operation results in a direct cost avoidance and eliminates the waste disposal environmental charge associated with that stripping operation.

In order to determine the cost of copper plating and stripping, manufacturers of gears and copper plating facilities were contacted. They were asked to determine the plating and stripping cost for a one inch thick, six-inch diameter gear with a one-inch diameter through hole. It was determined that the cost of stripping copper plating from a plated gear is equal to the cost of plating. Additionally, an environmental charge for the disposal of the waste created in the copper stripping process is also incurred.

The estimated cost reduction and avoidance is identified in Table 16. The project required an expenditure of $505,000 to complete. The projected savings (see Table 16) offset the project cost within a period of six months of pervasive implementation.

Table 16 – Cost benefit analysis (CBA). All dollar values are per year.

Cost Benefit Analysis		
Military Helicopter Spares and Replacements (See Table 15)	Total Number of Gears in Current Helicopter Fleet (4250 helicopters x 52 gears/helicopter)	221,000
	Total Number of Spare and Replacement Gears per Year (10% of above)[-]	22,100
	Gears Using Selective Copper Plating (80% of above)[*]	17,680
	Reduced Plating Thickness Savings ($10.50/gear)[**]	$185,640
	Copper Stripping Process Elimination ($8/gear)[+]	$141,440
	Re-plating Process Elimination ($8/gear)[+]	$141,440
	Elimination of One Grit Blast Operation ($2/gear)[+]	$35,360
	Waste Disposal Savings (Copper Related) 3.5%[+]	$16,398
	Subtotal Savings	*$520,278 per year*
Estimate for Gear Production Facility Similar in Size to Honeywell (Phoenix), Accessory Gear Output	Carburized Parts Per Year (Honeywell 1997 Numbers)	38,000
	Reduced Plating Thickness Savings ($10.50/gear)[**]	$399,000
	Parts using Selective Copper Plating (30% of 38,000)[**]	11,400
	Copper Stripping Process Elimination ($8/gear)[+]	$91,200
	Re-plating Process Elimination ($8/gear)[+]	$91,200
	Elimination of One Grit Blast Operation ($2/gear)[+]	$22,800
	Waste Disposal Charge Avoidance 3.5%[+]	$20,349
	Subtotal Savings	*$624,549 per year*
	Total Savings	*$1,144,827 per year*

[-] Conservative estimate based on [FC92].
[*] Engineering estimate.
[**] Original Honeywell estimate.
[+] Estimate based on industry survey.

Industry-Wide Implementation Plan

In order to implement the copper reduction process change and maximize the benefits that can be achieved, 102 gear manufacturers and heat treat facilities that process gears were contacted. Of the number contacted, 21 companies requested that the findings of the project be sent to them. These manufacturers are identified in Table 17.

In the discussions with the gear manufacturers, the minimum effective plating thickness requirement was noted and its application to each particular gear design. In order to ensure that the minimum thickness is applied in all the required areas of the gear design, individual gear design reviews would have to be made. Each review would address the result of the plating operation on areas such as holes and recesses that generally achieve less-thick plating than more exposed areas. The required plating thickness must be determined for exposed gear areas while ensuring the minimum effective plating is achieved in “hard to plate” areas of the gear. The cost of the finished ground aerospace gear makes the design review prior to implementation a cost-effective approach to avoiding unnecessary costly scrap while maximizing the cost savings achieved.

After the design review of the selected gear to be copper plated, several gear sets would be processed and evaluated to ensure that the process control can be maintained. Upon obtaining acceptable results, the process could be incorporated into the manufacturing procedure.

So, implementation of the low copper technique would require the following:

1) Individual gear design reviews to determine if thin copper was adequate to stop-off carbon would need to be conducted. This would mostly be a function of gear geometry and making sure that adequate copper would be plated in areas such as holes and recesses. Also, these parts must not be required to be subjected to a subcritical annealing operation.
2) Austenitizing furnaces would need to be qualified for the process to make sure that an adequate endothermic atmosphere could be maintained and no excess oxygen was present. Control of carbon potential between the levels of 0.65% and 0.95% at 1525°F should be sufficient to achieve this. To maintain optimum carbon in the surface, a carbon potential of 0.85% would be preferred.
3) Testing of candidate gears in a production environment would need to be performed to verify the ability of the process.
4) Once verification was complete, modifications of existing manufacturing processes for the copper plating operation prior to carburization, would need to be made. Also, the overall manufacturing process would need modified to eliminate grit blasting (cleaning), stripping and re-plating after carburization.

Implementation may also require mechanical tests such as rolling/sliding contact fatigue (R/SCF) testing before implementation is feasible. This would depend on individual

manufacturer's requirements (See the section “Recommendations/Future Work” on page 65).

Table 17 – Manufacturers that responded positively to receiving information regarding the Low Copper project. (continued on next page)

Company	Internet URL	Physical Address	Phone Numbers
Aero Gear	www.aerogear.com	1050 Day Hill Road Windsor, CT 06095	Voice: (860)688-0888 Fax: (860)285-8514
Arrow Gear	www.arrowgear.com	2301 Curtiss Street Downers Grove, IL 60515	Voice: (630)969-7640 Fax: (630)969-0253
Bell helicopter	www.bellhelicopter.textron.com	P.O. Box 482 Fort Worth, TX 76101	Voice: (817)280-3601 Fax: (817)280-3291
Boeing	www.boeing.com	5000 E. McDowell Rd. Mesa, AZ 85215	Voice: (480)891-3000 Fax: N/A
Boeing	www.boeing.com	Ridley Township, PA 19033	Voice: (610)591-2121 Fax: N/A
Boeing	www.boeing.com	Seattle, WA 98101	Voice: (206)544-1264 Fax: N/A
Boeing	www.boeing.com	4200 Southeast Blvd., Wichita, KS 67210	Voice: (316)526-2121 Fax: N/A
Chicago Gear Works, Inc.	www.chicagogearworks.com	1805 S. 55th Street Cicero, IL 60650	Voice: (800)343-3652 Fax: (800)432-7957
Derlan Precision Gear	www.derlan.com	6006 W. 73rd St. Bedford Park, IL 60638-6106	Voice: (708)728-2000 Fax: (708)728-2009
Foote-Jones/ Illinois Gear	www.footejones.com	2102 N. Natchez Ave. Chicago, IL 60707-3493	Voice: (773)622-8000 Fax: (773)622-8176
Forest City Gear Co.	www.fcgear.com	11715 Main St. Roscoe, IL 61073	Voice: (815)623-2168 Fax: (815)623-6620

Table 17 – Manufacturers that responded positively to receiving information regarding the Low Copper project. (concluded)

Company	Internet URL	Physical Address	Phone Numbers
Ingersoll Rand Company	www.ingersoll-rand.com	100 Main Street Athens, PA 18810	Voice: (570)888-7777 Fax: N/A
ITW Spiroid	www.itwspiroid.com	3700 West Lake Ave. Glenview, IL 60025	Voice: (800)253-7940 Fax: (847)657-5098
Productigear, Inc.	www.productigear.com	1900 W. 34th St. Chicago, IL 60608	Voice: (773)847-4505 Fax: (773)847-6348
Progressive Steell Treating	www.geartechnology.com/copage/progress.htm	922 Lawn Drive Loves Park, IL 61111	Voice: (815)877-2571 Fax: (815)877-7922
The Purdy Corp.	www.purdytransmissions.com	586 Hilliard St. Manchester, CT 06040	Voice: (860)649-0000 Fax: (860)645-6293
Reliance Gear Corp.	www.reliancegear.com	205 Factory Rd. Addison, IL 60101	Voice: (630)543-6640 Fax: (630)543-0520
Riley Gear Co.	www..rileygear.com	One Precision Drive St. Augustine, FL 32092	Voice: (904)829-5652 Fax: (904)829-5838
Rockford Heat Treaters, Inc.	N/A	2510 20th St. Rockford, IL 61104	Voice: (815)398-6533 Fax: N/A
Rolls-Royce Corp.	www.rolls-royce.com	P.O. Box 420 Indianapolis, IN 46206	Voice: (317)230-2000 Fax: (317)230-4020
Southwest United	www.swunited.com	422 S. St.Louis Ave. Tulsa, OK 74120	Voice: (918)587-4161 Fax: (918)582-6158

Overall Conclusions

Overview

The main conclusion drawn from the analyses performed on the DOE 1 specimens was that carbon did not penetrate the copper layers of plating of thicknesses between 0.001 inch to 0.0005 inch for carburization cycles between 1650°F and 1750°F.

DOE 2 showed that 0.0001 inch copper plating failed in an SAE/AISI 9310H heat treatment cycle, while 0.0005 inch plating performed as required. Secondary conclusions were that the factors of rotary furnace carbon potential, time in the rotary furnace, and quench transfer time did not play a significant role in the performance of the copper plating. Neither did these factors play a significant role in the measured hardness on the carburized end of each specimen.

Based on analysis of variance performed on the measured hardness of the plated halves of DOE 3 sample numbers 1 through 48, its accompanying main effects, secondary effects and pareto plots, the subcritical anneal at 1175°F was the most detrimental factor influencing failure of the plating. The macroetch performed on the samples supported this conclusion.

The analysis performed on the DOE 3 specimens that were plated after carburization indicated that any effects present were hidden by error. All main effects were within 1.5 HRC (converted from HK) of each other, well within the error expected when examining hardness using the Knoop method (see Appendix X1 and X2 of ASTM E384).

The analysis performed on the non-plated halves of the DOE 3 specimens indicated that all main effects were within 0.5 HRC. Again any effects were hidden by error in the measurements.

Cost Savings

The cost-benefit analysis performed showed that substantial industry-wide cost savings could be achieved by implementation of the "low copper" technique. And, in fact, projected savings based on pervasive implementation could offset the cost of the project within six months. Strictly looking at military helicopter replacement and spare manufacturing, savings offset the cost of the project within a period of one year.

Recommendations / Future Work

The thin copper plating technique for selective carburization needs to be tested on actual parts. This could be done using methods similar to those described in this report. Testing a part with a more advanced geometry than those tested here would allow the evaluation of current-density (end and corner) effects in the copper plating bath. It would also serve to better qualify the process from the standpoint of defect control in production copper plating and any size effect encountered in the heat treatment process.

Mechanical tests, such as rolling-sliding contact fatigue (R/SCF) tests, need to be performed on parts processed with thin copper to qualify the process for aerospace-quality parts. This would validate the process for both mechanical and wear characteristics of the final part.

Lastly, this technique needs to be tested using different types of copper plating methods such as electroless or non-cyanide. Waste streams from cyanide-based copper plating baths are expensive to treat and present environmental concerns. The additional cost of electroless and non-cyanide baths due to the better process control they require would also need to be evaluated.

System Validation Report

The system validation report for this project is contained in the Honeywell preproduction experimentation report (Appendix H).

Monograph

No monograph was formally required or prepared for this project.

Period of Performance

The period of performance for this project was from June 14, 1999 to March 28, 2001. A finalized task plan was sent to the sponsor on July 8, 1999.

Research Approach

The following tasks were performed:

1. Project Management: IITRI executed project management encompassing project planning, scheduling, resource management, purchasing, reporting, technology transfer, project review presentations, and technical training. IITRI prepared and submitted to AMCOM the required data and deliverables. IITRI provided the required detailed accounts of the schedule, cost, and technical performance of the project during the INFAC Quarterly Reviews. The purpose of these reviews was to provide a detailed account of the schedule, cost, and technical performance of the project. These reviews were also used to present issues, technical alternatives, accomplishments, and detailed plans for the remaining effort.
2. Literature Search: A literature search was conducted as part of this project. Keywords were developed and then searched for in various databases to find material that may aid development of the project. A “References” section has been included near the end of this report.
3. Experimentation was performed in three sub-experiments, referred to as DOE 1, DOE 2, and DOE 3. DOE, here, is an acronym for *D*esign *o*f *E*xperiment. Designed experiments are experiments that are run for a minimum of cost, but have the ability to provide the analyst with the maximum amount of information available.
4. Analysis: Analysis was performed using DOE techniques. Results include ANOVA (Analysis of Variance), main effects plots, secondary effects plots (interaction plots), and tertiary effects plots (cube plots).
5. End of Project Briefing: An End of Project briefing will be conducted after this contract has expired, as agreed to by the sponsor and IITRI management.
6. Business Analysis: A cost/benefit analysis of this project was performed after the experimentation had concluded. Honeywell has also performed a cost benefit analysis of implementation of the thin copper technique for their facility, see Appendix I.
7. Final Report: This final report was prepared to present the review procedure, the results, and the business analysis. This final report details the advantages and limitations of the technique developed. Implementation issues at IITRI's subcontractor are discussed.

Data and Deliverables

1. Data: With this final report IITRI has delivered all data in accordance with the Contract Data Requirement List (CDRL) DD Form 1423 that is part of the INFAC contract.
2. Project Plan: Within 30 days after the contract award, IITRI prepared a task plan according to DI-MGMT-80909, to provide technical, schedule, cost, and other related project data. The plan included a detailed project milestone chart covering all the major activities of the project, major subcontracts, and equipment/material purchases and a labor loading/estimated-cost-to-be-incurred for each major task
3. Status Report: IITRI has prepared, in IITRI's format, a periodic written Status Report as required by the INFAC contract, according to DI-MGMT-80368. These reports contained the project's objectives and procedures, status of technical progress against project plan, technical issues, data, results obtained to date, and the effort planned for the next reporting period. These reports were included in the INFAC Project Status Report every four weeks. In addition, IITRI provided to the Government, cost/schedule status, billing, and payment status.
4. Preliminary Cost/Benefit Analysis: IITRI submitted a preliminary cost/benefit analysis within 60 days after the kick off meeting.
5. End of Project Briefing and Demonstration: At the conclusion of the technical effort, IITRI prepared viewgraphs for a presentation that will be performed after the close of the contract.
6. Final Report: IITRI prepared and submitted this final report. IITRI shall distribute this final report in accordance with a distribution list prepared by the Government.

With this final report all required data and deliverables required by the INFAC contract have been prepared and submitted.

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Appendix A

State of the Art Review

Selective Case Hardening in the Aerospace Industry: A State of the Art Review

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Introduction

The manufacture of precision gears is an expensive task. Raw stock must be machined into a gear blank. Teeth are cut, turning the blank into what one would generally regard as a gear. The gear must then be heat treated to impart hardness and other necessary physical properties to it. Finally, the gear must be finished to ensure that proper geometric tolerances are met. These tasks are tedious and lengthy. It can take anywhere from a few days to a few weeks to turn raw stock into a gear.

Heat treatment alone is an expensive and lengthy process, involving many subtasks. Precision gears are made of steel, principally SAE/AISI 9310H[6,7]. The heat treatment task could be broken down briefly as NQT[8,9], carburizing, slow cooling, austenitizing, quenching, deep freezing, and tempering. This paper will examine a detail of the carburization and reaustenitizing processes, viz. copper plating used as a carbon diffusion barrier. Cost associated with this facet of heat treatment is significant and worthy of study.

Copper plating is applied to the surface of a precision gear to selectively carburize it. Selective carburization is the process by which certain areas of a surface are carburized, while others are not. This may be done because additional machining may be needed on non-carburized areas after carburizing [GM89]. It is also possible that a tough, fracture-

[6] SAE/AISI 9310H has been the primary material used to produce precision gears for years. A newer material of interest is Pyrowear Alloy 53 ™. Pyrowear Alloy 53 is a much more expensive alloy, but has been gaining wider acceptance in the aerospace industry due to better properties at elevated temperature.

[7] SAE/AISI 9310H has also been known by the names AISI 9310, AMS 6260, or AMS 6265. These alloys have essentially the same composition, but are produced in different fashions.

[8] NQT, normalizing quenching and tempering, is a process by which different heats of steel can be made to have the same initial properties [Heberling 1990]. Properties resulting from heat treatment are not path independent. NQT is most easily described as a method by which to start a new path.

[9] The use of NQT is not ubiquitous among manufacturers, and debate continues on the value of its use.

resistant surface may be required in some areas of the finished part when in service. In either instance, copper plating is applied to areas that are not to be carburized.

This State of the Art Review (SOAR) is a result of both a literature search and an industry review in which we visited three U.S. precision gear manufacturers and interviewed engineers and other personnel involved in heat treatment and copper plating operations.

Relationship of Carburizing to Precision Gears

The primary use of selectively carburized precision gears[10] is in helicopter transmissions. The environment that a gear needs to withstand inside a transmission of this type is one of heat, friction and stress. The material used to accomplish this task is chosen on the basis of these factors. The material must withstand high temperature[11] without significant loss of properties. This is in conflict with the fact that most materials lose strength when their temperature is raised. The materials must also have a wear resistant surface that does not experience a significant lowering of properties at elevated temperature. This surface must have good surface fatigue[12] resistance if it is expected to have a long service life. On top of all these requirements, these gears must be able to handle extremely high loads. Fortunately, certain alloys of steel exhibit these desired properties when properly manufactured.

The process of carburizing adds a wear-resistant and surface fatigue-resistant surface to steel. Carbon can be diffused, at a high enough temperature, into a steel surface. The surface can then be quenched in oil and tempered in a furnace. The existence of carbon alone in a steel surface such as by the diffusion described, but subsequently cooled slowly rather than quenched, will result in a relatively fragile structure of ferrite and cementite, not nearly what is required of a transmission component. Quenching the part in oil after austenitizing will result in a structure composed mostly of martensite. Martensite is very hard, which translates to good wear resistance. It is, however, far too brittle to have adequate fatigue resistance. The act of tempering the martensitic structure lowers the hardness slightly[13], but increases fatigue resistance.

Austenitizing for Hardening

Austenitizing is the process of bringing steel to a temperature[14] that causes its structure to transform to that of austenite[15]. The steel is austenitized in a controlled atmosphere furnace,

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[10] A precision gear is most easily defined as a gear of exceptional quality, quantitatively meeting the requirements of an AGMA quality number 12 gear or higher.

[11] A good estimate of the operating temperature of a helicopter transmission is between 225ºF and 275ºF.

[12] Surface fatigue is caused by the repeated application and removal of load [Drago 1988].

[13] The word 'slightly' is used, but the actual loss of hardness can be negligible in practice.

[14] The austenitizing temperature of pure carbon steel (i.e., no alloying elements) with 0.1% carbon is approximately 1425ºF. For comparison, the austenitizing temperature of SAE/AISI 9310H is 1500 °F.

usually a rotary hearth furnace, and is then either free quenched or die quenched. One may ask why the step of austenitizing is performed in the first place rather than quenching directly after carburizing. There are at least two reasons for this.

Quenching directly after carburizing has been explored as an alternative to carburizing, slow cooling, reaustenitizing, and quenching [Ker88]. The major advantage of this process is labor and energy savings due to the reduced number of steps needed to attain a carburized and quenched surface. An additional advantage observed is less distortion due to the absence of a second heating operation. A major disadvantage is the coarser microstructure developed as a result of directly quenching the part. Direct quenching also tends to cause the formation of microcracks in the structure. These microcracks can lead to early failure of the part. Retained austenite can also be a problem in direct quenching. Large amounts of retained austenite can reduce rolling contact fatigue performance significantly; Kern has stated that the decrease in performance can be as much as 40%.

Direct quenching of precision gears would be difficult to perform on parts that require press quenching[16]. Carburizing is typically performed as a batch process in a large furnace. The furnace is generally of the pit or integral-quench type. Transfer of parts to a press quenching fixture is painstaking at best. One must also consider the effect of exposing a carburized surface, at austenitizing temperatures, to an air atmosphere for an extended period of time. Case loss can be a problem. Free quenching parts in an integral-quench furnace is an easier task, but still faces the difficulties mentioned above.

Because of the reasons given above and partly because of precedence, reheat quenching has been the predominant process performed on precision gears in the aerospace industry. Kern's paper [Ker88] documents the performance of SAE/AISI 9310H as a result of direct quenching, but no full-scale experiment is cited to substantiate the reported performance.

Copper Deposition

Copper deposition onto steel has traditionally been accomplished by the method of cyanide bath electrodeposition. The actual steps followed vary from manufacturer to manufacturer, but the basic process can be broken down as follows [Sri83]:

- Mask component where plating is not desired.
- Degrease in an alkali solution for approximately 15 minutes at roughly 90°C.
- Rinse in water.

[15] Austenite is a phase of steel that is stable at high temperature. Its crystal structure is that of face-centered cubic (FCC). It is also referred to as gamma iron (γ-iron).

[16] Press quenching is a process in which a component is rapidly taken from a furnace where its temperature was above the austenitizing temperature, placed in a mechanically operated fixture, and simultaneously quenched with oil and held in place by applied pressure. This process is also variously referred to as 'fixture quenching', and 'die quenching' for obvious reasons.

- Rinse in 25 to 30% hydrochloric acid for 5 to 8 minutes at room temperature.
- Rinse in water.
- Rinse in 1% sodium cyanide for 2 to 9 minutes.
- Cyanide copper electrodeposit[17].
- Rinse in water.
- De-mask components.
- Rinse in water.
- Degrease in alkali solution.
- Dry components.

Though electrodeposition in and of itself is a simple process, settings of voltage, amperage, bath pH, and bath composition will have an effect on the plating quality, adhesion, and speed. Preparation and cleaning of components are accomplished by various methods, but do not differ greatly from the sequence stated. Cleaning operations performed after electrodeposition is a function of mask geometry and visual inspection.

Plating is occasionally performed in two steps. First, a 'strike' solution is employed that deposits copper very quickly. Only a thin coating can be applied by this method. A longer, slower, electrodeposit[18] is then used for depositing a thicker coating. At least one manufacturer reported that they strike[19] steel surfaces in an iterative fashion to promote faster deposit in the thicker-plating bath.

Inspections performed on the coating include an appearance inspection, an adhesion test, porosity measurement, and thickness inspection. The appearance of the coating should be free from visual defects such as blisters, pits, and stains (ASTM B734). The adhesion test can be performed on a shim. The shim is bent and inspected visually for cracking and/or flaking of the copper plating. Porosity measurement is performed by microscopically inspecting the surface of the copper or by macroscopically applying a ferroxl[20] solution and inspecting for blue spots[21]. Thickness[22] inspection is performed using an eddy current

[17] Bath variables include voltage, amperage, time, and temperature.

[18] This copper electrodeposit occurs in a bath with composition, voltage, and amperage different from that in the strike solution.

[19] In this sense, 'strike' refers to the act of depositing a thin coating of copper. The usual thickness of the strike coating is between 0.0001 and 0.0003 inches.

[20] See [ASTM B734], Appendix X1 for an explanation of this test.

[21] Blue spots would indicate exposed iron at the surface.

[22] Thickness is inspected on flat or critical surfaces only. The electrodeposition process will deposit thicker coatings at locations with high current density, such as corners, and thinner coatings at notches, where a drop in current density can occur. Part of the reason that thick (0.001 to 0.003 inch)

device (ASTM E376), a magnetic thickness gauge (ASTM B499), or by metallographic inspection. These tests are usually performed on an as needed basis [23] and do not rely on SPC techniques [24]. Close control of plating bath parameters has produced consistent results at many manufacturers.

Stripping of the copper plating is done, generally, in the following manner:

- Grit blasting.
- Degreasing.
- Water rinse.
- Immersion in a stripping solution such as chromic acid or an alkaline solution.
- Water rinse.
- Blow or still air dry.

Copper can be recovered from the stripping bath and the solution can be reused, but the task is usually outsourced.

Aerospace manufacturers have experimented with non-cyanide based plating baths with mixed results. The consensus seems to be that it can be applied successfully with plating thicknesses up to about 0.001 inches. Thicknesses greater than 0.001 inches tend to produce unacceptable amounts of porosity. Though most manufacturers interviewed for this paper stated that they have tried applying non-cyanide copper, it was unclear if they have run parts through their furnaces to examine diffusion barrier performance. They also said that bath composition and maintenance were major factors in determining successful copper deposition.

Diffusion Barriers for Carburizing and Austenitizing

In the process of selective carburizing, copper plating is used as a barrier to prevent carbon from diffusing into the steel substrate. Gears going into a carburizing cycle will typically be copper plated to 0.001 to 0.003 inches. The carburizing atmosphere will protect the integrity of the plated surface but any exposure to an air atmosphere at carburizing temperatures can quickly strip the surface of at least 0.0001 to 0.0002 inches of plating, due to oxidation.

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coatings have traditionally been applied to parts going into a selective carburizing operation is to ensure that a minimum thickness is present on plated surfaces. Two of the inspection methods mentioned in the text, viz. eddy current and magnetic thickness measurement devices, are difficult to apply to notches and corners, and consequently are primarily utilized on flat surfaces.

[23] Generally, a visual inspection is performed 100% of the time. If a component fails this test, additional tests are performed and plating baths are inspected.

[24] Manufacturers did express a concern over the fact that most of them do not have a SPC system for their copper plating inspection operations.

Copper plated surfaces[25] can be divided into two groups based on surface location in relation to free steel surface and copper plating mask. The junction of free steel surface and copper mask is illustrated in Figure 1(a). When the area of interest is far from a free surface / copper mask junction (FCJ), carbon will diffuse one-dimensionally into the steel substrate. When the area of interest is at the FCJ, carbon diffuses two-dimensionally, and will diffuse to approximately the carburizing depth under the mask in a radial fashion with the center at the FCJ [GM89], this is illustrated in Figure 1(b). Obviously, three dimensions are involved in more complicated geometries.

Another common method of masking[26] components for selective carburization involves the use of stop-off paint. Paint, consisting of refractory substances and a binder of glass, can be applied to the surface of the component where carbon is to be stopped off[27] [Nov74]. Manufacturers of commonly used paints generally advise the consumer to apply the paint liberally, to approximately a certain thickness[28]. Those manufacturers do warn, however, that even a small amount applied to areas not to be stopped off will almost certainly be so. Even a finger print or glance of an application brush can have serious ramifications on final part quality if such actions deposit paint on the wrong area of a component. The paint is also difficult to apply consistently to a large number of parts. Geometric tolerances on areas to be carburized are difficult enough to maintain on an individual part, let alone many.

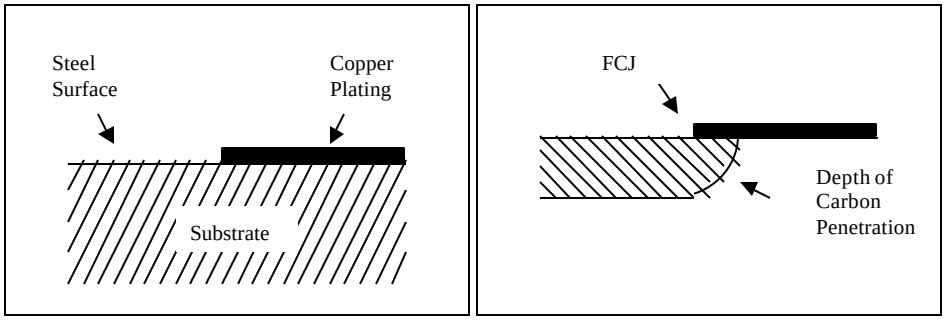

Figure 1 – (a) The surface of a steel component that has been copper plated. The geometry shown illustrates the area where the copper plating meets the steel surface. This geometry is idealized; the

[25] The following sentences would apply to other diffusion barriers in this geometry, not just copper plating.

[26] Be careful with the use of the term "masking". It is used liberally in this paper to refer to either the coating of copper (or stop-off paint) on the steel surface, or the "masking" that is done to apply the copper or stop-off coating itself.

[27] Carbon is prevented from diffusing into the substrate.

[28] Manufacturer specific.

actual junction is much more complex. (b) Carbon penetration into a steel surface at the FCJ is two dimensional.

An additional problem with stop-off paints is the fact that they cannot be used with press quenching. Recall that copper plating is used in conjunction with press quenching in the austenitization procedure to prevent escape of carbon from the case. In order for press quenching to be successfully implemented, a consistently thick layer of paint would need to be applied, which, as was stated in the preceding paragraph, is highly improbable.

A painted-on diffusion barrier is thicker than a copper plated one. However, tolerances in a press quench need to be preserved or improved, neither of which can be accomplished with a thicker layer. Thinner layers allow for greater dimensional accuracy in the press quenching process and provide more consistent gears.

Stop-off paint liquefies at just below carburizing temperatures. This produces a fairly uniform surface on the areas to which it was applied, however, as a consequence of being liquid, the paint has a tendency to drip and spread over areas that were to be carburized.

Removal of copper plating after carburizing or quenching is routine and of little trepidation. Removal of stop-off paint is generally messy, prone to producing part damage, and is labor consuming. It is commonly done with glass bead.

Manufacturers expressed the concern that certain stop-off paints contain free metals, such as tin, that may diffuse into the steel substrate at high temperatures and promote embrittling of the steel. As mentioned, however, components of stop-off paint are typically refractories and are therefore in such a state as to be chemically stable. Be that as it may, it was still a concern to manufacturers. No study that the author knows of examines surface reactions between steel and stop-off paint to prove or disprove the argument.

Given the variables and consequences stated in the preceding paragraphs, the preference among aerospace manufacturers is to use copper plating rather than a stop-off paint for selective carburizing or austenitizing. It is not uncommon practice, however, to use stop-off paint to make minor repairs to damaged or poor quality copper plating. Highly damaged or very poor copper plating will usually be re-plated.

Conclusions

The current state of the art in selective carburizing has not changed much, if at all, in recent years. The following points summarize the current practices at major U.S. aerospace manufacturers.

- Copper cyanide bath plating is the application method of choice for copper plating. Non-cyanide plating baths have been examined in house at aerospace manufacturers with success only at plating thicknesses below 0.001 inches.
- Current practice is to apply copper used as a diffusion barrier during carburizing to thicknesses between 0.001 and 0.003 inches. The reasons for the thickness

range chosen are partly due to specification and partly due to in-house precedent.

- There is a greater variety of thicknesses of copper used as diffusion barriers during austenitizing than there is used during carburizing, viz. on the lower tolerance. Most manufacturers would rather plate to the high end of the tolerance rather than the low end due to the high cost of scrap should the plating fail. This practice does reduce dimensional accuracy in the press quench. However, it should be noted that of the aerospace manufacturers visited, the thinner coatings were generally applied to shallower cased parts.
- Stop-off paint is not a viable alternative to copper plating when press quenching of parts is needed. Additionally, part damage as a result of poor application or removal practices is common.

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Appendix B

DOE 1 Raw Surface Roughness Data

This section contains the raw data from the surface roughness measurements of DOE 1. These data were generated using a TSK profilometer. All reported values are R_a and are in μinch.

Table B1 - Raw surface roughness data from DOE 1 samples after creating rough surfaces and prior to copper plating. (Obs. = Observation)}

	R_a on As-received End (μin)				R_a on Machined End (μin)			
Sample	Obs. 1	Obs. 2	Obs. 3	Average	Obs. 1	Obs. 2	Obs. 3	Average
1	42.88	45.93	45.80	44.87	118.02	116.07	115.94	116.68
2	56.77	53.10	52.89	54.25	120.61	119.29	120.52	120.14
3	49.58	45.96	58.84	51.46	124.09	118.61	120.67	121.12
4	64.34	52.49	33.16	50.00	120.84	120.02	117.47	119.44
5	50.08	36.36	41.65	42.70	118.33	119.21	120.37	119.30
6	47.14	37.85	44.22	43.07	118.70	119.04	120.98	119.57
7	55.62	52.72	52.62	53.65	103.10	97.90	95.04	98.68
8	27.42	37.34	27.42	30.73	95.27	96.09	96.76	96.04
9	43.37	48.09	37.32	42.93	98.17	95.37	96.78	96.77
10	40.07	51.83	56.84	49.58	104.23	97.90	95.63	99.25
11	25.13	23.16	25.68	24.66	98.87	100.00	103.66	100.84
12	44.47	48.43	43.35	45.42	98.51	96.81	97.06	97.46
13	52.12	47.70	48.76	49.53	95.14	97.75	95.18	96.02
14	41.28	44.02	57.71	47.67	95.33	97.33	99.40	97.35
15	45.25	51.61	45.81	47.56	93.69	94.91	94.77	94.46
16	39.64	53.32	43.36	45.44	94.67	97.16	97.38	96.40
17	32.56	48.23	45.70	42.16	96.15	98.61	98.51	97.76
18	58.59	46.17	54.67	53.14	97.84	97.94	96.23	97.34
19	46.33	40.31	45.52	44.05	95.93	96.41	98.90	97.08
20	39.02	43.00	39.57	40.53	97.19	101.28	97.88	98.78
21	41.11	45.98	51.37	46.15	102.06	99.62	100.40	100.69
22	46.59	41.79	44.45	44.28	104.51	98.81	98.64	100.65
23	42.72	46.97	61.11	50.27	97.12	97.88	100.32	98.44

24	56.15	58.42	41.70	52.09	99.46	98.47	96.67	98.20

Table B2 - Raw surface roughness data from DOE 1 samples after copper plating. (Obs. = Observation)

	R_a on As-received End (μin)				R_a on Machined End (μin)			
Sample	Obs. 1	Obs. 2	Obs. 3	Average	Obs. 1	Obs. 2	Obs. 3	Average
1	52.38	53.41	38.92	48.24	120.10	118.27	116.51	118.29
2	50.99	44.67	56.77	50.81	124.64	123.28	124.22	124.05
3	54.65	52.07	57.81	54.84	123.99	122.31	122.65	122.98
4	52.74	52.83	60.93	55.50	127.60	131.37	127.58	128.85
5	47.97	44.05	46.48	46.17	122.35	125.99	122.62	123.65
6	44.28	37.75	50.81	44.28	132.08	129.40	128.41	129.96
7	50.83	50.01	50.17	50.34	97.23	94.69	105.93	99.28
8	41.92	35.86	34.38	37.39	100.71	101.97	102.34	101.67
9	50.54	43.01	48.04	47.20	108.22	106.61	107.82	107.55
10	47.60	51.80	51.14	50.18	101.99	104.01	104.00	103.33
11	26.78	27.28	26.02	26.69	101.04	98.24	97.85	99.04
12	49.22	53.31	48.05	50.19	96.67	99.65	101.65	99.32
13	48.74	56.41	59.49	54.88	95.55	96.14	99.44	97.04
14	48.87	52.79	42.76	48.14	102.24	106.85	102.67	103.92
15	45.97	52.93	56.99	51.96	101.72	101.91	100.08	101.24
16	45.83	40.92	42.89	43.21	100.38	97.61	95.80	97.93
17	46.97	50.59	38.62	45.39	99.31	99.11	100.93	99.78
18	50.38	50.81	41.16	47.45	99.14	99.66	98.12	98.97
19	39.30	48.08	46.62	44.67	100.78	105.65	99.13	101.85
20	43.33	49.03	50.24	47.53	108.65	102.66	109.14	106.82
21	50.60	51.33	46.80	49.58	102.19	102.57	100.64	101.80
22	44.74	57.44	56.22	52.80	98.20	99.54	94.68	97.47
23	40.95	31.33	33.13	35.14	103.95	95.37	93.91	97.74
24	50.70	62.64	47.57	53.64	100.97	99.32	99.87	100.05

Appendix C
DOE 1 Raw Mass Data

This section contains the raw data from the mass measurements of DOE 1.

Table C1 - Raw mass data from the DOE 1 samples. The mass of each sample was measured at each of the three process points indicated. All values are in grams.

Sample Number	Before Plating	After Plating	Difference (Mass gain after plating)	After Carburizing	Difference (Mass gain after carburizing)
1	42.63	42.66	0.03	42.67	0.01
2	42.68	42.83	0.15	42.83	0.00
3	42.67	42.70	0.03	42.72	0.02
4	42.61	42.77	0.16	42.77	0.00
5	42.63	42.77	0.14	42.77	0.00
6	42.61	42.77	0.16	42.78	0.01
7	42.64	42.67	0.03	42.67	0.00
8	42.61	42.78	0.17	42.78	0.00
9	42.61	42.78	0.17	42.78	0.00
10	42.50	42.66	0.16	42.66	0.00
11	42.44	42.48	0.04	42.48	0.00
12	42.47	42.62	0.15	42.62	0.00
13	42.57	42.60	0.03	42.61	0.01
14	42.48	42.66	0.18	42.66	0.00
15	42.56	42.73	0.17	42.73	0.00
16	42.63	42.78	0.15	42.78	0.00
17	42.55	42.58	0.03	42.60	0.02
18	42.60	42.63	0.03	42.64	0.01
19	42.68	42.82	0.14	42.82	0.00
20	42.51	42.68	0.17	42.68	0.00
21	42.47	42.62	0.15	42.62	0.00
22	39.13	39.16	0.03	39.17	0.01
23	39.10	39.24	0.14	39.25	0.01

24	39.16	39.31	0.15	39.31	0.00

Appendix D

DOE 2 Fully Nested ANOVA

This was generated using MiniTab version 12.23

Fractional Factorial Fit

Estimated Effects and Coefficients for PE@D<=0. (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		47.27	0.4419	106.96	0.000
Block		-0.27	0.4419	-0.62	0.548
CPT	-23.77	-11.88	0.4419	-26.89	0.000
RFCP	-1.03	-0.52	0.4419	-1.17	0.261
TIRF	1.44	0.72	0.4419	1.63	0.123
QTT	0.38	0.19	0.4419	0.43	0.672
CPT*RFCP	0.37	0.18	0.4419	0.42	0.682
CPT*TIRF	-2.66	-1.33	0.4419	-3.01	0.009
CPT*QTT	1.43	0.72	0.4419	1.62	0.126
RFCP*TIRF	-0.42	-0.21	0.4419	-0.47	0.642
RFCP*QTT	0.44	0.22	0.4419	0.50	0.623
TIRF*QTT	0.09	0.05	0.4419	0.11	0.917
CPT*RFCP*TIRF	0.53	0.27	0.4419	0.60	0.557
CPT*RFCP*QTT	0.39	0.20	0.4419	0.45	0.662
CPT*TIRF*QTT	0.39	0.20	0.4419	0.45	0.662
RFCP*TIRF*QTT	-1.54	-0.77	0.4419	-1.75	0.101
CPT*RFCP*TIRF*QTT	0.51	0.25	0.4419	0.57	0.575

Analysis of Variance for PE@D<=0. (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	1	2.37	2.37	2.37	0.38	0.548
Main Effects	4	4545.97	4545.97	1136.49	181.88	0.000
2-Way Interactions	6	76.97	76.97	12.83	2.05	0.121
3-Way Interactions	4	23.80	23.80	5.95	0.95	0.461
4-Way Interactions	1	2.05	2.05	2.05	0.33	0.575
Residual Error	15	93.73	93.73	6.25		
Total	31	4744.89				

Estimated Coefficients for PE@D<=0. using data in uncoded units

Term	Coef
Constant	85
Block	-0
CPT	-53535
RFCP	-29
TIRF	-0
QTT	-1
CPT*RFCP	5859
CPT*TIRF	-109
CPT*QTT	738
RFCP*TIRF	0
RFCP*QTT	1
TIRF*QTT	0

```
CPT*RFCP*TIRF          -190
CPT*RFCP*QTT           -773
CPT*TIRF*QTT            -16
RFCP*TIRF*QTT            -0
CPT*RFCP*TIRF*QTT        21
```

Estimated Effects and Coefficients for CE@D<=0. (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		62.2812	0.2479	251.21	0.000
Block		-0.4125	0.2479	-1.66	0.117
CPT	0.7500	0.3750	0.2479	1.51	0.151
RFCP	0.8125	0.4062	0.2479	1.64	0.122
TIRF	0.3375	0.1687	0.2479	0.68	0.506
QTT	0.5000	0.2500	0.2479	1.01	0.329
CPT*RFCP	0.0250	0.0125	0.2479	0.05	0.960
CPT*TIRF	-0.4000	-0.2000	0.2479	-0.81	0.432
CPT*QTT	-0.2875	-0.1437	0.2479	-0.58	0.571
RFCP*TIRF	1.7625	0.8813	0.2479	3.55	0.003
RFCP*QTT	-0.3500	-0.1750	0.2479	-0.71	0.491
TIRF*QTT	0.5250	0.2625	0.2479	1.06	0.306
CPT*RFCP*TIRF	-0.5500	-0.2750	0.2479	-1.11	0.285
CPT*RFCP*QTT	-0.0625	-0.0313	0.2479	-0.13	0.901
CPT*TIRF*QTT	0.8125	0.4063	0.2479	1.64	0.122
RFCP*TIRF*QTT	-0.3000	-0.1500	0.2479	-0.61	0.554
CPT*RFCP*TIRF*QTT	-0.2375	-0.1188	0.2479	-0.48	0.639

Analysis of Variance for CE@D<=0. (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	1	5.4450	5.4450	5.4450	2.77	0.117
Main Effects	4	12.6925	12.6925	3.1731	1.61	0.222
2-Way Interactions	6	29.9825	29.9825	4.9971	2.54	0.067
3-Way Interactions	4	8.4525	8.4525	2.1131	1.07	0.404
4-Way Interactions	1	0.4513	0.4513	0.4513	0.23	0.639
Residual Error	15	29.5050	29.5050	1.9670		
Total	31	86.5288				

Estimated Coefficients for CE@D<=0. using data in uncoded units

Term	Coef
Constant	75.4
Block	-0.4
CPT	289.1
RFCP	-19.2
TIRF	-0.3
QTT	0.1
CPT*RFCP	12656.2
CPT*TIRF	2.3
CPT*QTT	-677.3
RFCP*TIRF	0.4
RFCP*QTT	-0.1
TIRF*QTT	-0.0
CPT*RFCP*TIRF	-161.5
CPT*RFCP*QTT	515.6
CPT*TIRF*QTT	11.8
RFCP*TIRF*QTT	0.0
CPT*RFCP*TIRF*QTT	-9.9

Estimated Effects and Coefficients for CE@D>0.0 (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		62.5375	0.2345	266.66	0.000

Block		-0.9000	0.2345	-3.84	0.002
CPT	0.9750	0.4875	0.2345	2.08	0.055
RFCP	-0.2125	-0.1062	0.2345	-0.45	0.657
TIRF	1.3750	0.6875	0.2345	2.93	0.010
QTT	1.0500	0.5250	0.2345	2.24	0.041
CPT*RFCP	0.1625	0.0812	0.2345	0.35	0.734
CPT*TIRF	-0.5750	-0.2875	0.2345	-1.23	0.239
CPT*QTT	0.2500	0.1250	0.2345	0.53	0.602
RFCP*TIRF	1.7875	0.8938	0.2345	3.81	0.002
RFCP*QTT	0.3125	0.1563	0.2345	0.67	0.515
TIRF*QTT	0.5750	0.2875	0.2345	1.23	0.239
CPT*RFCP*TIRF	-0.9875	-0.4938	0.2345	-2.11	0.053
CPT*RFCP*QTT	-0.4625	-0.2312	0.2345	-0.99	0.340
CPT*TIRF*QTT	0.8750	0.4375	0.2345	1.87	0.082
RFCP*TIRF*QTT	-0.0125	-0.0063	0.2345	-0.03	0.979
CPT*RFCP*TIRF*QTT	-1.0875	-0.5437	0.2345	-2.32	0.035

Analysis of Variance for CE@D>0.0 (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	1	25.920	25.920	25.920	14.73	0.002
Main Effects	4	31.911	31.911	7.978	4.53	0.013
2-Way Interactions	6	32.344	32.344	5.391	3.06	0.037
3-Way Interactions	4	15.639	15.639	3.910	2.22	0.116
4-Way Interactions	1	9.461	9.461	9.461	5.38	0.035
Residual Error	15	26.400	26.400	1.760		
Total	31	141.675				

Estimated Coefficients for CE@D>0.0 using data in uncoded units

Term	Coef
Constant	73
Block	-1
CPT	20102
RFCP	-18
TIRF	-0
QTT	1
CPT*RFCP	-10781
CPT*TIRF	-613
CPT*QTT	-2007
RFCP*TIRF	0
RFCP*QTT	-1
TIRF*QTT	-0
CPT*RFCP*TIRF	536
CPT*RFCP*QTT	2141
CPT*TIRF*QTT	42
RFCP*TIRF*QTT	0
CPT*RFCP*TIRF*QTT	-45

* NOTE * There is partial confounding, no alias table was printed.

Fully Nested Analysis of Variance

Analysis of Variance for PE@D<=0.

Source	DF	SS	MS	F	P
CPT	1	4519.6278	4519.6278	942.018	0.001
RFCP	2	9.5956	4.7978	0.250	0.790
TIRF	4	76.7812	19.1953	3.589	0.059
QTT	8	42.7925	5.3491	0.891	0.546
Error	16	96.0950	6.0059		
Total	31	4744.8922			

Variance Components

Source	Var Comp.	% of Total	StDev
CPT	282.177	96.75	16.798
RFCP	-1.800*	0.00	0.000
TIRF	3.462	1.19	1.861
QTT	-0.328*	0.00	0.000
Error	6.006	2.06	2.451
Total	291.644		17.078

* Value is negative, and is estimated by zero.

Expected Mean Squares

1 CPT	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2) + 16.00(1)
2 RFCP	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2)
3 TIRF	1.00(5) + 2.00(4) + 4.00(3)
4 QTT	1.00(5) + 2.00(4)
5 Error	1.00(5)

Analysis of Variance for CE@D<=0.

Source	DF	SS	MS	F	P
CPT	1	4.5000	4.5000	1.703	0.322
RFCP	2	5.2863	2.6431	0.359	0.719
TIRF	4	29.4625	7.3656	4.779	0.029
QTT	8	12.3300	1.5413	0.706	0.683
Error	16	34.9500	2.1844		
Total	31	86.5287			

Variance Components

Source	Var Comp.	% of Total	StDev
CPT	0.116	3.09	0.341
RFCP	-0.590*	0.00	0.000
TIRF	1.456	38.76	1.207
QTT	-0.322*	0.00	0.000
Error	2.184	58.15	1.478
Total	3.757		1.938

* Value is negative, and is estimated by zero.

Expected Mean Squares

1 CPT	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2) + 16.00(1)
2 RFCP	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2)

```
 3 TIRF      1.00(5) +  2.00(4) +  4.00(3)
 4 QTT       1.00(5) +  2.00(4)
 5 Error     1.00(5)


Analysis of Variance for CE@D>0.0

Source     DF             SS           MS       F       P
CPT         1         7.6050       7.6050  26.568   0.036
RFCP        2         0.5725       0.2862   0.022   0.978
TIRF        4        51.1325      12.7831   3.404   0.066
QTT         8        30.0450       3.7556   1.149   0.385
Error      16        52.3200       3.2700
Total      31       141.6750


Variance Components

Source      Var Comp.   % of Total       StDev
CPT            0.457         7.35       0.676
RFCP          -1.562*        0.00       0.000
TIRF           2.257        36.24       1.502
QTT            0.243         3.90       0.493
Error          3.270        52.51       1.808
Total          6.227                    2.495

* Value is negative, and is estimated by zero.
```

Expected Mean Squares

1	CPT	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2) + 16.00(1)
2	RFCP	1.00(5) + 2.00(4) + 4.00(3) + 8.00(2)
3	TIRF	1.00(5) + 2.00(4) + 4.00(3)
4	QTT	1.00(5) + 2.00(4)
5	Error	1.00(5)

Appendix E

DOE 3 Fully Nested ANOVA for Plated Halves of Samples 1 – 48 (Plated Prior to Carburizing)

This was generated using MiniTab version 12.23

Fractional Factorial Fit

Estimated Effects and Coefficients for HRC (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		43.822	0.2357	185.89	0.000
Copper P	-1.981	-0.991	0.2357	-4.20	0.001
Case Dep	-1.069	-0.534	0.2357	-2.27	0.038
Subcriti	7.706	3.853	0.2357	16.34	0.000
Rotary F	0.369	0.184	0.2357	0.78	0.446
Copper P*Case Dep	-1.819	-0.909	0.2357	-3.86	0.001
Copper P*Subcriti	-1.719	-0.859	0.2357	-3.65	0.002
Copper P*Rotary F	-0.981	-0.491	0.2357	-2.08	0.054
Case Dep*Subcriti	-2.481	-1.241	0.2357	-5.26	0.000
Case Dep*Rotary F	-1.344	-0.672	0.2357	-2.85	0.012
Subcriti*Rotary F	-0.269	-0.134	0.2357	-0.57	0.577
Copper P*Case Dep*Subcriti	-1.381	-0.691	0.2357	-2.93	0.010
Copper P*Case Dep*Rotary F	0.181	0.091	0.2357	0.38	0.706
Copper P*Subcriti*Rotary F	-0.469	-0.234	0.2357	-0.99	0.335
Case Dep*Subcriti*Rotary F	-0.206	-0.103	0.2357	-0.44	0.668
Copper P*Case Dep*Subcriti *Rotary F	-0.181	-0.091	0.2357	-0.38	0.706

Analysis of Variance for HRC (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	4	516.719	516.719	129.180	72.64	0.000
2-Way Interactions	6	122.074	122.074	20.346	11.44	0.000
3-Way Interactions	4	17.624	17.624	4.406	2.48	0.086
4-Way Interactions	1	0.263	0.263	0.263	0.15	0.706
Residual Error	16	28.455	28.455	1.778		
Pure Error	16	28.455	28.455	1.778		
Total	31	685.135				

Unusual Observations for HRC at 0

Obs	HRC at 0	Fit	StDev Fit	Residual	St Resid
8	46.1000	43.9000	0.9430	2.2000	2.33R
24	41.7000	43.9000	0.9430	-2.2000	-2.33R

R denotes an observation with a large standardized residual

Estimated Coefficients for HRC using data in uncoded units

Term	Coef
Constant	2
Copper P	56007
Case Dep	567

Subcriti	0
Rotary F	46
Copper P*Case Dep	-788263
Copper P*Subcriti	17
Copper P*Rotary F	-70823
Case Dep*Subcriti	-0
Case Dep*Rotary F	-666
Subcriti*Rotary F	0
Copper P*Case Dep*Subcriti	-10
Copper P*Case Dep*Rotary F	972460
Copper P*Subcriti*Rotary F	-1
Case Dep*Subcriti*Rotary F	0
Copper P*Case Dep*Subcriti *Rotary F	-828

Alias Structure

I
Copper
Case
Subcriti
Rotary
Copper*Case
Copper*Subcriti
Copper*Rotary
Case*Subcriti
Case*Rotary
Subcriti*Rotary
Copper*Case*Subcriti
Copper*Case*Rotary
Copper*Subcriti*Rotary
Case*Subcriti*Rotary
Copper*Case*Subcriti*Rotary

Fully Nested Analysis of Variance

Analysis of Variance for HRC at 0

Source	DF	SS	MS	F	P
Copper P	1	31.4028	31.4028	1.764	0.315
Case Dep	2	35.6006	17.8003	0.126	0.885
Subcriti	4	563.2388	140.8097	42.609	0.000
Rotary F	8	26.4375	3.3047	1.858	0.139
Error	16	28.4550	1.7784		
Total	31	685.1347			

Variance Components

Source	Var Comp.	% of Total	StDev
Copper P	0.850	2.25	0.922
Case Dep	-15.376*	0.00	0.000
Subcriti	34.376	91.02	5.863
Rotary F	0.763	2.02	0.874
Error	1.778	4.71	1.334

```
Total         37.768                    6.146

* Value is negative, and is estimated by zero.

Expected Mean Squares

 1 Copper P  1.00(5) +  2.00(4) +  4.00(3) +  8.00(2) + 16.00(1)
 2 Case Dep  1.00(5) +  2.00(4) +  4.00(3) +  8.00(2)
 3 Subcriti  1.00(5) +  2.00(4) +  4.00(3)
 4 Rotary F  1.00(5) +  2.00(4)
 5 Error     1.00(5)
```

Appendix F

DOE 3 Fully Nested ANOVA for Plated Halves of Samples 49 – 96 (Plated After Carburizing)

This was generated using MiniTab version 12.23

Fractional Factorial Fit

Estimated Effects and Coefficients for HRC (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		63.7500	0.3480	183.17	0.000
Copper P	-1.4875	-0.7438	0.3480	-2.14	0.048
Case Dep	0.4125	0.2063	0.3480	0.59	0.562
Subcriti	-0.0875	-0.0437	0.3480	-0.13	0.902
Rotary F	-0.2125	-0.1062	0.3480	-0.31	0.764
Copper P*Case Dep	0.4500	0.2250	0.3480	0.65	0.527
Copper P*Subcriti	-0.0250	-0.0125	0.3480	-0.04	0.972
Copper P*Rotary F	-0.3000	-0.1500	0.3480	-0.43	0.672
Case Dep*Subcriti	-0.0500	-0.0250	0.3480	-0.07	0.944
Case Dep*Rotary F	-0.5750	-0.2875	0.3480	-0.83	0.421
Subcriti*Rotary F	-0.2000	-0.1000	0.3480	-0.29	0.778
Copper P*Case Dep*Subcriti	0.0375	0.0188	0.3480	0.05	0.958
Copper P*Case Dep*Rotary F	-0.1375	-0.0687	0.3480	-0.20	0.846
Copper P*Subcriti*Rotary F	-0.2375	-0.1187	0.3480	-0.34	0.737
Case Dep*Subcriti*Rotary F	-0.3375	-0.1687	0.3480	-0.48	0.634
Copper P*Case Dep*Subcriti *Rotary F	-1.1500	-0.5750	0.3480	-1.65	0.118

Analysis of Variance for HRC (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	4	19.485	19.485	4.8713	1.26	0.327
2-Way Interactions	6	5.330	5.330	0.8883	0.23	0.961
3-Way Interactions	4	1.525	1.525	0.3812	0.10	0.981
4-Way Interactions	1	10.580	10.580	10.5800	2.73	0.118
Residual Error	16	62.020	62.020	3.8763		
Pure Error	16	62.020	62.020	3.8763		
Total	31	98.940				

Observations for HRC at 0

Obs	HRC at 0	Fit	StDev Fit	Residual	St Resid
1	63.3000	63.8500	1.3922	-0.5500	-0.40
2	63.3000	63.0500	1.3922	0.2500	0.18
3	64.9000	65.1500	1.3922	-0.2500	-0.18
4	63.9000	63.1500	1.3922	0.7500	0.54
5	64.4000	64.6500	1.3922	-0.2500	-0.18
6	61.4000	61.9000	1.3922	-0.5000	-0.36
7	65.9000	64.1500	1.3922	1.7500	1.26
8	64.4000	64.9500	1.3922	-0.5500	-0.40
9	64.9000	65.1500	1.3922	-0.2500	-0.18
10	60.5000	62.2000	1.3922	-1.7000	-1.22
11	62.4000	63.9500	1.3922	-1.5500	-1.11

12	64.4000	63.8500	1.3922	0.5500	0.40
13	64.9000	64.4000	1.3922	0.5000	0.36
14	61.4000	63.1500	1.3922	-1.7500	-1.26
15	63.9000	64.6500	1.3922	-0.7500	-0.54
16	57.7000	61.8000	1.3922	-4.1000	-2.95R
17	64.4000	63.8500	1.3922	0.5500	0.40
18	62.8000	63.0500	1.3922	-0.2500	-0.18
19	65.4000	65.1500	1.3922	0.2500	0.18
20	62.4000	63.1500	1.3922	-0.7500	-0.54
21	64.9000	64.6500	1.3922	0.2500	0.18
22	62.4000	61.9000	1.3922	0.5000	0.36
23	62.4000	64.1500	1.3922	-1.7500	-1.26
24	65.5000	64.9500	1.3922	0.5500	0.40
25	65.4000	65.1500	1.3922	0.2500	0.18
26	63.9000	62.2000	1.3922	1.7000	1.22
27	65.5000	63.9500	1.3922	1.5500	1.11
28	63.3000	63.8500	1.3922	-0.5500	-0.40
29	63.9000	64.4000	1.3922	-0.5000	-0.36
30	64.9000	63.1500	1.3922	1.7500	1.26
31	65.4000	64.6500	1.3922	0.7500	0.54
32	65.9000	61.8000	1.3922	4.1000	2.95R

R denotes an observation with a large standardized residual

Estimated Coefficients for HRC using data in uncoded units

Term	Coef
Constant	22
Copper P	99336
Case Dep	892
Subcriti	0
Rotary F	62
Copper P*Case Dep	-2111751
Copper P*Subcriti	-182
Copper P*Rotary F	-148957
Case Dep*Subcriti	-1
Case Dep*Rotary F	-1228
Subcriti*Rotary F	-0
Copper P*Case Dep*Subcriti	3955
Copper P*Case Dep*Rotary F	2951341
Copper P*Subcriti*Rotary F	241
Case Dep*Subcriti*Rotary F	2
Copper P*Case Dep*Subcriti *Rotary F	-5251

Alias Structure

I
Copper
Case
Subcriti
Rotary
Copper*Case
Copper*Subcriti
Copper*Rotary
Case*Subcriti
Case*Rotary

```
Subcriti*Rotary
Copper*Case*Subcriti
Copper*Case*Rotary
Copper*Subcriti*Rotary
Case*Subcriti*Rotary
Copper*Case*Subcriti*Rotary
```

Fully Nested Analysis of Variance

Analysis of Variance for HRC at 0

Source	DF	SS	MS	F	P
Copper P	1	17.7013	17.7013	11.875	0.075
Case Dep	2	2.9812	1.4906	61.154	0.001
Subcriti	4	0.0975	0.0244	0.012	1.000
Rotary F	8	16.1400	2.0175	0.520	0.824
Error	16	62.0200	3.8763		
Total	31	98.9400			

Variance Components

Source	Var Comp.	% of Total	StDev
Copper P	1.013	19.97	1.007
Case Dep	0.183	3.61	0.428
Subcriti	-0.498*	0.00	0.000
Rotary F	-0.929*	0.00	0.000
Error	3.876	76.41	1.969
Total	5.073		2.252

* Value is negative, and is estimated by zero.

Expected Mean Squares

```
 1 Copper P  1.00(5) +  2.00(4) +  4.00(3) +  8.00(2) + 16.00(1)
 2 Case Dep  1.00(5) +  2.00(4) +  4.00(3) +  8.00(2)
 3 Subcriti  1.00(5) +  2.00(4) +  4.00(3)
 4 Rotary F  1.00(5) +  2.00(4)
 5 Error     1.00(5)
```

Appendix G

DOE 3 Fully Nested ANOVA for Unplated Halves of Samples 1 through 96

This was generated using MiniTab version 12.23

Factorial Design

Full Factorial Design

Factors:	4	Base Design:	4, 16
Runs:	64	Replicates:	4
Blocks:	none	Center pts (total):	0

All terms are free from aliasing

Fractional Factorial Fit

Estimated Effects and Coefficients for HRC (coded units)

Term	Effect	Coef	StDev Coef	T	P
Constant		63.1797	0.2439	259.01	0.000
Plating	0.4406	0.2203	0.2439	0.90	0.371
Case Dep	0.4719	0.2359	0.2439	0.97	0.338
Subcriti	-0.3281	-0.1641	0.2439	-0.67	0.504
Rotary F	0.4531	0.2266	0.2439	0.93	0.358
Plating*Case Dep	0.3531	0.1766	0.2439	0.72	0.473
Plating*Subcriti	0.6656	0.3328	0.2439	1.36	0.179
Plating*Rotary F	-0.5531	-0.2766	0.2439	-1.13	0.263
Case Dep*Subcriti	-0.3156	-0.1578	0.2439	-0.65	0.521
Case Dep*Rotary F	0.7656	0.3828	0.2439	1.57	0.123
Subcriti*Rotary F	0.6781	0.3391	0.2439	1.39	0.171
Plating*Case Dep*Subcriti	1.0781	0.5391	0.2439	2.21	0.032
Plating*Case Dep*Rotary F	-0.4156	-0.2078	0.2439	-0.85	0.398
Plating*Subcriti*Rotary F	-0.7156	-0.3578	0.2439	-1.47	0.149
Case Dep*Subcriti*Rotary F	-0.0344	-0.0172	0.2439	-0.07	0.944
Plating*Case Dep*Subcriti* Rotary F	-0.2031	-0.1016	0.2439	-0.42	0.679

Analysis of Variance for HRC (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	4	11.677	11.677	2.9192	0.77	0.552
2-Way Interactions	6	32.310	32.310	5.3849	1.41	0.229
3-Way Interactions	4	29.574	29.574	7.3936	1.94	0.119
4-Way Interactions	1	0.660	0.660	0.6602	0.17	0.679
Residual Error	48	182.783	182.783	3.8080		
Pure Error	48	182.783	182.783	3.8080		

```
Total                63     257.004

Unusual Observations for HRC at 0

Obs   HRC at 0        Fit  StDev Fit   Residual   St Resid
 13    54.2000    59.9500     0.9757    -5.7500      -3.40R
 22    57.8000    62.0000     0.9757    -4.2000      -2.49R

R denotes an observation with a large standardized residual

Estimated Coefficients for HRC using data in uncoded units

Term                            Coef
Constant                        71.1
Plating                         -1.8
Case Dep                      -130.1
Subcriti                        -0.0
Rotary F                      -11.7
Plating*Case Dep                12.2
Plating*Subcriti                -0.0
Plating*Rotary F                 3.6
Case Dep*Subcriti               -0.0
Case Dep*Rotary F              201.3
Subcriti*Rotary F                0.0
Plating*Case Dep*Subcriti        0.1
Plating*Case Dep*Rotary F      -45.7
Plating*Subcriti*Rotary F       -0.0
Case Dep*Subcriti*Rotary F      -0.0
Plating*Case Dep*Subcriti*
Rotary F                        -0.1


* NOTE * There is partial confounding, no alias table was printed.
```

Fully Nested Analysis of Variance

```
Analysis of Variance for HRC at 0

Source     DF             SS           MS       F       P
Plating     1         3.1064       3.1064   1.118   0.401
Case Dep    2         5.5578       2.7789   0.383   0.704
Subcriti    4        29.0031       7.2508   1.587   0.268
Rotary F    8        36.5537       4.5692   1.200   0.319
Error      48       182.7825       3.8080
Total      63       257.0036


Variance Components

Source      Var Comp.   % of Total       StDev
Plating         0.010         0.24       0.101
Case Dep      -0.279*         0.00       0.000
Subcriti        0.335         7.72       0.579
Rotary F        0.190         4.38       0.436
Error           3.808        87.67       1.951
Total           4.344                    2.084
```

```
* Value is negative, and is estimated by zero.

Expected Mean Squares

 1 Plating   1.00(5) +  4.00(4) +  8.00(3) + 16.00(2) + 32.00(1)
 2 Case Dep  1.00(5) +  4.00(4) +  8.00(3) + 16.00(2)
 3 Subcriti  1.00(5) +  4.00(4) +  8.00(3)
 4 Rotary F  1.00(5) +  4.00(4)
 5 Error     1.00(5)
```

Appendix H

Honeywell Preproduction Experimentation Final Report